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RCRA QUALITY ASSURANCE PROJECT PLAN SOLID WASTE MANAGEMENT UNITS 8, 15,
18, 19, 20, (SWMU 8) (SWMU 15) (SWMU 18) (SWMU 19) (SWMU 20) OLD GUN TUB
STORAGE LOT UNEXPLODED ORDNANCES 5 AND 7 (UXO 5) (UXO 7) BUILDING 2044
DROP TOWER/TEST RAIL SITE AND RANGES NSA CRANE IN
3/1/2007
TETRA TECH

CONTRACT NUMBER N62467-04-D-0055



**Resource Conservation and Recovery Act
Addendum No. 2 to the
Quality Assurance Project Plan
for
SWMUs 8, 15, 18, 19, 20 and The Old Gun Tub
Storage Lot
for
UXO 5 (Building 2044 Drop Tower/Test Rail Site)
and UXO 7 (Ranges)**

**Naval Surface Warfare Center
Crane Division
Crane, Indiana**

Contract Task Order 0034

March 2007



Southeast

2155 Eagle Drive

North Charleston, South Carolina 29406

**RESOURCE CONSERVATION AND RECOVERY ACT
ADDENDUM NO. 2 TO THE
QUALITY ASSURANCE PROJECT PLAN
FOR
SWMUs 8, 15, 18, 19, 20, AND THE OLD GUN TUB STORAGE LOT
FOR
UXO 5 (BUILDING 2044 DROP TOWER/TEST RAIL SITE) AND
UXO 7 (RANGES)**

**NAVAL SURFACE WARFARE CENTER
CRANE DIVISION
CRANE, INDIANA**

**COMPREHENSIVE LONG-TERM
ENVIRONMENTAL ACTION NAVY (CLEAN) CONTRACT**

**Submitted to:
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ACRONYMS

%R	Percent Recovery
°C	Degrees Celsius
BC/BC	Big Clifty-Beech Creek
bgs	below ground surface
CAAA	Crane Army Ammunition Activity
CAD	Cartridge Actuated Device
CFR	Code of Federal Regulations
CLEAN	Comprehensive Long-Term Environmental Action Navy
CLP	Contract Laboratory Program
CSM	Conceptual Site Model
CTO	Contract Task Order
DE	Depositional Environment
DQI	Data Quality Indicator
DQO	Data Quality Objectives
EI	Environmental Indicator
FOL	Field Operations Leader
GC	Gas Chromatography/Mass Spectrometry
HASP	Health and Safety Plan
HPLC	High Performance Liquid Chromatography
HSM	Health and Safety Manager
IDEM	Indiana Department of Environmental Management
IDL	Instrument Detection Limit
IDW	Investigation Derived Waste
IM	Interim Measure
IMWP	Interim Measures Work Plan
MC	Munitions Constituents
MDL	Method Detection Limit
MEC	Munitions and Explosives of Concern
mL	Milliliters
MRP	Munitions Response Program
MS	Matrix Spike
MSD	MS Duplicate
msl	mean sea level

NAD	North American Datum
NAVD	North American Vertical Datum
NAVFAC SE	Naval Facilities Engineering Command Southeast
NFA	No Further Action
NFESC	Naval Facilities Engineering Service Center
NSWC	Naval Surface Warfare Center
OB	Open Burning
OE	Ordinance and Explosive
OGTSL	Old Gun Tub Storage Lot
OPR	Old Pistol Range
ORR	Old Rifle Range
OSHA	Occupational Safety and Health Administration
PA	Preliminary Assessment
PAD	Propellant Actuated Device
PCB	Polychlorinated biphenyls
PE	Performance Evaluation
PM	Project Manager
PPE	Personal Protective Equipment
ProgMan	Program Manager
PTA	Pyrotechnic Test Area
QA	Quality Assurance
QAC	Quality Assurance Coordinator
QAM	Quality Assurance Manager
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QC	Quality Control
RCRA	Resource Conservation and Recovery Act
RCRIS	Resource Conservation and Recovery Act Information System
RFA	RCRA Facility Assessment
RFI	RCRA Facility Investigation
RI	Remedial Investigation
RL	Reporting Limit
RPD	Relative Percent Difference
RPM	Remedial Project manager
SAP	Sample and Analysis Plan
SOP	Standard Operating procedure

SOW	Statement of Work
SSO	Site Safety Officer
SWMU	Solid Waste Management Unit
TAL	Target Analyte List
TOM	Task Order Manager
TiNUS	Tetra Tech NUS, Inc.
TV	Threshold Value
U.S. EPA	United States Environmental Protection Agency
USACE	United States Army Corps of Engineers
USCS	Unified Soil Classification System
UTL	Upper Tolerance Level
UXO	Unexploded Ordnance
XRF	X-Ray Fluorescence

1.0 PROJECT DESCRIPTION

The Naval Surface Warfare Center (NSWC) Crane prepared a Quality Assurance Project Plan (QAPP) [Tetra Tech NUS, Inc. (TtNUS), 2004] for a Phase III Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI) at the following Solid Waste Management Units (SWMUs):

- SWMU 8, Building 106 Pond
- SWMU 15, Roads and Grounds Area

That QAPP also governed data collection for completion of the United States Environmental Protection Agency (USEPA) Region 5 RCRA Conservation and Recovery Information System (RCRIS) code Environmental Indicator (EI) Form (Form CA725) for the following sites:

- SWMU 18, Load and Fill Area Buildings
- SWMU 19, Pyrotechnic Test Area (PTA)
- SWMU 20, Crane Army Ammunition Activity (CAAA) Quality Assurance (QA)/Quality Control (QC) Test Area
- Old Gun Tub Storage Lot (OGTSL)

That QAPP was approved by U.S. EPA Region 5, and the required field work was conducted in accordance with the QAPP.

QAPP Addendum No. 1 (TtNUS, 2006) was developed to include Interim Measures (IMs) Work Plans (IMWPs) to govern IMs conducted at the following Crane sites:

- Old Pistol Range (OPR) and Old Rifle Range (ORR) at SWMU 7
- SWMU 8, Building 106 Pond Area
- SMWU 13, Mine Fill B
- SWMU 17, PCB Pole Yard

The focus of the IMWPs was excavation of contaminated soil and sediment and other IMs that were designed to reduce or eliminate the extent and magnitude of contamination at each of these four sites. QAPP Addendum No. 1, dated November 2006, was approved by U.S. EPA Region 5, and the required field work was conducted in accordance with the approved QAPP.

This QAPP Addendum No. 2 presents the project organization, objectives, planned activities, and specific QA/QC procedures associated with sample collection and analysis for the RCRA Facility Assessment (RFA) of two sites, UXO 5 and UXO 7, under the Navy Munitions Response Program (MRP). All applicable portions of the previous QAPP and QAPP Addendum No. 1 that are not superseded by this QAPP Addendum No. 2 have been reviewed to ensure that they are still valid.

1.1 DESCRIPTION OF QAPP ADDENDUM NO. 2

This QAPP Addendum No. 2 will govern the sampling and analyses of surface soils to provide data on potentially site-related organic and inorganic chemical concentrations at UXO 5 and UXO 7. Upon completion of RFA activities at UXO 5, an RFA Report will be submitted to the Navy with recommendations for future activities. UXO 7 is comprised of individual areas within the boundaries of SWMU 7 in which several past investigations have occurred. Upon the completion of the RFA at UXO 7, the results will be incorporated into the SWMU 7 RFI Report and will be submitted to the Navy. The report will include formal human health and ecological risk assessments.

The specific objectives to be achieved during each RFA include the following:

UXO 5

- Based on sampling in areas where metal contamination would be present if released to the environment, determine whether further study of UXO 5 in the form of an RFI is warranted to delineate the contamination and estimate risks to human health or the environment.

UXO 7

- Based on sampling in areas where metal contamination (ie: lead) would be present if released to the environment, determine whether further study of UXO 7 in the form of an RFI is warranted to delineate the contamination and estimate risks to human health or the environment.
- If RFA data indicate that an RFI is warranted, before moving to the RFI, determine whether the RFI can be avoided by using RFA data to assess whether human health or ecological risks are low enough not to require further study.

This QAPP Addendum No. 2 was prepared for the NSWC Crane facility located in Crane, Indiana through the Naval Facilities Engineering Command Southeast (NAFVAC SE) under Contract Task Order (CTO) 0034 for the Comprehensive Long-Term Environmental Action Navy (CLEAN) IV, Contract Number

N62467-04-D-0055. This QAPP Addendum No. 2 generally does not repeat information already provided in the approved QAPP. Figure 1-1 shows the general location of NSWC Crane, and Figure 1-2 shows the general locations of UXO 5 and UXO 7. Figure 1-3 shows the specific location of UXO 5. Figure 1-4 shows the individual sites that make up UXO 7. Site-specific geologic and hydrogeologic information for UXO 5 is provided based on the preliminary site assessment. Site-specific geologic and hydrogeologic information for UXO 7 is provided based on previous site investigations and the RFI Report for SWMU 7 in which UXO 7 is a sub area. For any work to be conducted under this QAPP Addendum No. 2 that is unique to this project, the appropriate information is presented in this document or references are provided to other documents containing the required information.

This QAPP Addendum No. 2 consists of five sections: Section 1.0 is this introduction, which includes descriptions of the QAPP, site descriptions, and the conceptual site models (CSMs) for each UXO site. Section 2.0 includes the project organization and responsibilities. Section 3.0 includes the Sampling and Analysis Plan (SAP). Section 4.0 includes Laboratory Operations and Data Reduction, Review, and Management. Section 5.0 includes all references. The appendices included in this QAPP Addendum No. 2 contain the following information:

- Appendix A - Field Standard Operating Procedures
- Appendix B - Laboratory Standard Operating Procedures on compact disk (CD)
- Appendix C - Instruction Manual for the INNOV-X-SYSTEMS X-Ray Fluorescence Spectrometer

1.2 NSWC SITE DESCRIPTION

Refer to Section 1.0 of the original QAPP for information regarding the site description of NSWC Crane.

1.3 UXO 5 SITE DESCRIPTION

This section is a presentation of background information, general site characteristics, and physical site characteristics specific to UXO 5 and UXO 7 that are the focus of these RFAs.

1.3.1 Operations

UXO 5 [Building 2044 (B-2044) Drop Tower/Test Rail Site] is located adjacent to Highway 304 and consists of two areas, the drop tower and test rail. The test area is located east of Building 2044 and is located in an area that is maintained by periodic mowing. UXO 5 is bordered to the north, south, and east by woods and to the west by Highway 45. The actual test site currently consists of a drop tower approximately 100 feet tall with an 8- by 8-foot concrete pad on the southwestern side of the base of the

tower. Periodically, 20-mm cartridges were dropped from the top of the tower onto the concrete pad to verify that cartridges would not detonate if mishandled. Therefore it is assumed that a relatively small proportion (i.e., fewer than 1 percent) of the 20-mm cartridges would have actually detonated. Approximately 75 feet southeast of the drop tower is a test rail approximately 97 feet in length from east and west. The test rail was used to test cartridge actuated devices (CADs) and propellant actuated devices (PADs) in ejection seats. CADs and PADs are items which release precise explosive or propellant energy to perform controlled work functions in a variety of applications. The site was used from January 1951 through December 1983, with average actual site usage reported to be approximately 9 days per month.

An MRP preliminary assessment (PA) has been conducted, and no evidence was observed indicating munitions and explosives of concern (MEC) on the ground surface at this site. During the testing periods, it was Navy policy to remove all retrievable MEC after testing. Subsurface MEC and surface/subsurface MEC have not been investigated; however, based on the operations conducted at the site, no surface or subsurface MEC is expected. Munitions constituents (MCs) that may be present would consist of trace explosives and metals near the drop tower, test rail, and possibly in the nearby grassy drainageway. Based on the known testing operations conducted at the site, the MCs, if present, would be primarily in the surface soil. Site features for UXO 5 are presented on Figure 1-3.

The Final Preliminary Assessment indicates that black powder and black powder plus carboxy terminated polybutadiene (CTPB), N-53 were the primary MC of CADs and PADs tested at UXO 5 (Malcolm Pirnie, Inc, 2005). Though not explicitly identified as such, the CTPB appears to have been a binder that was added to the mixture to slow the combustion for a controlled burning of the MC. In this capacity its purpose would also have been to provide a uniform matrix throughout which the black powder was dispersed.

Black powder is comprised of potassium nitrate (approximately 75 percent by weight), charcoal (approximately 15 percent by weight), and sulfur (approximately 10 percent by weight). Potassium accounts for approximately 25 percent of the mass of black powder.

In addition to black powder, other propellants and explosives are often used in CADs and PADs to provide the energy that allows the device to function. Therefore, the list of explosives and propellants that are specific in SW-846 Method 8330 will also be analyzed.

Similar arguments can be made for the limited ability to detect any sulfur compounds released from black powder combustion. Furthermore, most sulfur compounds would most likely be transformed through

oxidation to sulfates and the naturally occurring sulfate concentration is unknown. Nevertheless, the amount of combustion products containing sulfur that are added to their native soil concentration will be less significant than for potassium compounds. All other black powder constituents remaining after total or partial combustion are viewed to be insignificant. Metals such as lead will be monitored to determine whether any significant masses of those metals are present as a result of site operations because some of the metals may have been released from projectiles or cartridge casings.

1.3.2 Topography

UXO 5 is located on a hilly area at an elevation of approximately 790 feet above mean sea level (msl). The area surrounding the site slopes upward to the northwest to an elevation of 810 feet above msl. To the southeast, the land slopes downward into a valley to an elevation of 750 feet above msl.

1.3.3 Geology

No wells or borings have been installed at the site; therefore, the site-specific geology of the site is unknown. A description of the regional geology can be found in the original QAPP (TtNUS, 2004).

1.3.4 Soil and Vegetation Types

UXO 5 is a lightly wooded area with young trees and sparse undergrowth. The area immediately surrounding the test rail and the drop tower is grass covered. The northern, southern, and eastern sides of the site are wooded. The distances from tree line to drop tower and test rail can be seen on Figure 1-3.

1.3.5 Hydrology

UXO 5 is located within the central portion of NSWC Crane and lies within the central drainage basin. Surface water runoff from the site drains south into an unnamed tributary, that flows southwest into Boggs Creek. Boggs Creek eventually empties into the East Fork of the White River south of NSWC Crane.

1.3.6 Hydrogeology

There are no wells on the site; consequently, the site-specific hydrogeology is unknown. However, a description of the regional hydrogeology can be found in the original QAPP (TtNUS, 2004).

1.3.7 UXO 5 Conceptual Site Model

The CSM for UXO 5 was developed following guidance documents issued by the U.S. EPA for hazardous waste sites and the United States Army Corps of Engineers (USACE) for ordnance and explosive (OE) sites. Guidance documents include the U.S. EPA's Guidance for Conducting Remedial Investigations and Feasibility Studies under CERCLA (EPA/540/G-89/004) and the USACE CSM Guidance and Development of Integrated Conceptual Site Models for Environmental Ordnance and Explosives (OE) Sites (2003).

The CSM describes the site and its environmental settings. The CSM presents information regarding: (1) MEC and/or MCs known or suspected to be at the site; (2) current and future reasonable anticipated or proposed uses of the real property; and (3) actual, potentially complete, or incomplete exposure pathways that link them. The CSM is the basis for future risk evaluation, prioritization, and remediation cost estimate.

The CSM is presented in a series of information profiles that present information about the site (see Table 1-1).

The configuration of the site is such that MC releases to surface soil from the drop tower are expected to be of small quantities. Because releases would occur through detonation after cartridges impacted the test pad, the contaminants would be released in a gaseous cloud near the ground surface at the base of the tower. The clouds would contain gaseous and particulate combustion products of explosives or propellants. Most of the gases would consist of nitrogen, water vapor, carbon dioxide, and perhaps nitrogen and sulfur oxides. The particulates may have contained explosives, propellants, and inorganic constituents such as metals or metal compounds. The clouds would not move far from the tower before returning to the ground surface because of their close proximity to this surface. Thus, the majority of contamination is expected to exist on the concrete pad and surrounding soil. Over time the contaminants would wash from the concrete pad onto adjacent surface soils. Eventually, the contaminants may have migrated through surface soil to deeper soils and perhaps as deep as the groundwater table. Surface soils may also have been washed by precipitation into nearby drainage channels. However, the majority of contamination is expected to reside near or in surface soils because of the deposition mechanism, and surface soils are expected to pose the most significant exposure route for humans and ecological receptors.

At the test rail, the deposition mechanism and exposure scenarios are similar to those at the drop tower. Therefore, surface soils at the test rail are expected to be the most contaminated medium and the most significant with regard to exposure of human and ecological receptors.

Over time, chemical and biochemical processes are expected to have acted on the organic compounds to reduce their concentrations. The processes that could occur include photolysis of chemicals exposed to sunlight, hydrolysis of chemicals in contact with water, dilution, advection, microbiological degradation, and other natural processes. The rates at which these processes would occur are unknown and are expected to vary widely from compound to compound and from one environmental condition to another.

1.3.8 UXO 5 Data Quality Objectives

The U.S. EPA Data Quality Objectives (DQO) process was used to develop the sampling design for collecting UXO 5 RFA data. The seven steps of the process are described briefly below.

1.3.8.1 Step 1 - PROBLEM DEFINITION

The following is the problem definition:

1. MCs and/or MEC may be present in surface soils although the presence of MEC is highly unlikely.
2. If MEC is detected during the RFA at UXO 5, it could present an acute hazard.
3. If found to be present in surface soils, MCs may have migrated deeper into subsurface soils, as well as groundwater, and any nearby sediment and surface water.
4. The CSM indicates that potentially complete pathways for human and ecological receptors both under current land uses and potential future land uses.
5. If present in significant concentrations in soils, MCs could present risks to human and ecological receptors.
6. This RFA is being conducted to determine whether conditions exist that would warrant further study.

1.3.8.2 Step 2 - DECISIONS TO BE MADE

The primary goal of this RFA at UXO 5 is to obtain environmental data for use in making the following decisions:

1. Determine whether MEC is present within the study area. If it is, initiate an immediate response to remove the associated acute hazard, otherwise no action for MEC is required.
2. Determine whether MCs are present within the study area in quantities or concentrations that require proceeding to a RFI/Corrective Measures Study (RFI/CMS). If they are, proceed to the RFI/CMS; otherwise do not investigate further.

1.3.8.3 Step 3 - INPUTS REQUIRED TO MAKE THE DECISION

Data and information that will be required to make these decisions includes the following:

1. Visual inspection for the presence of MEC in surface soil.
2. Concentrations in surface soils of SW-846 Method 8330 explosives.
3. Concentrations in surface soils of SW-846 Method 6010B metals to determine if Target Analyte List (TAL) metals are present in soils at concentrations greater than background and screening levels.
 - a. All UXO 5 surface soils belong to Soil Group 3 as defined in the NSWC Crane Basewide Soil Background Study (TtNUS, 2001). This knowledge will allow site and background soils to be matched so the data represent similar soils when conducting background comparisons.
 - b. Background lead data in soils similar to UXO 5 (from NSWC Crane Basewide Soil Background Study, January, 2001). The 95% Upper Tolerance Level (UTL) from the background study will be used for comparisons.
4. Screening levels for explosives, propellants, and TAL metals to determine if it is necessary to expand the study area and whether or not to proceed to an RFI.
5. Method detection limits less than screening levels.

1.3.8.4 Step 4 - DELINEATION OF STUDY BOUNDARY

1. The horizontal boundary is defined as the area where testing most likely took place, as shown on Figure 3-1.
2. MCs, if present, would be primarily in surface soils; therefore, the depth of interest is 0 to 2 feet bgs.
3. MEC, if present, would be primarily in the top 6 inches of surface soil. However, visual inspections of soils deeper than 6 inches will continue during sampling activities.

1.3.8.5 Step 5 - DEFINITION OF RULES FOR DECISION MAKING

1. If MEC is observed through visual inspections within or around the study area, stop work and take action to remove the acute hazard posed by the MEC; otherwise, if no MEC is observed, continue sampling.
2. If MCs are detected in concentrations in any soil sample greater than background concentrations (metals) and screening values (metals and organic MCs), evaluate potential risk, otherwise No Further Action (NFA).
3. If initial risk evaluation shows that a potentially unacceptable condition exists, proceed to an RFI, otherwise do not investigate further.

1.3.8.6 Step 6 - PERFORMANCE CRITERIA

1. Because this is an RFA and depends heavily on biased sampling, probability limits for false decision errors were not established.
2. Performance criteria for new analytical data (8330 explosives and TAL methods only) are presented in the original QAPP.

1.3.8.7 PLAN FOR OBTAINING THE DATA

Sample Collection and Analyses

1. Surface Soil
 - Surface soil samples will be collected at 0 to 2 feet bgs in the areas shown on Figure 3-1.

- Samples will be collected within each of the established grids [Standard Operating Procedure (SOP) CTO034-05].
- Because the soil samples are shallow, they can be readily collected via a hand auger.
- Unexploded ordnance (UXO) clearance is not required because the PA did not identify any concerns that MEC would be present. Nevertheless, sample collection will proceed with the understanding that MEC could be present and, if present, poses an acute hazard.
- Composite samples will be collected throughout the sample collection areas to obtain a more representative sample than would be afforded by grab samples considering the heterogeneity of soil contamination (reference Appendix A of proposed U.S. EPA SW-846 Method 8330B).

2. Analysis

- Surface soils will be analyzed for explosives (SW-846 8330) and TAL metals (SW-846 6010B).

3. Surface water and sediment are not present at the site; therefore, sampling of these media is not included for this RFA. Drainage ditch "sediment" will be treated as surface soil because the ditches are typically free of water.

4. Groundwater sample collection is not warranted at this time.

1.4 UXO 7 SITE DESCRIPTION

1.4.1 Operations

UXO 7 is located within the boundaries of the ORR, which occupies approximately 20 acres. The ORR is part of a larger unit designated as SWMU 7. UXO 7 is immediately west of NSWC Crane Highway 8 in the flat-lying floodplain of Turkey Creek. The site consists of a flat, grass-covered area bisected from north to south by an unnamed but maintained gravel road. This road provides access to various groundwater monitoring wells located within SWMU 7 and to a powder burning area that is a RCRA-permitted open burning (OB) facility. Currently, only the area of the ORR consisting of the OB facility is active. The other areas used for rifle and pistol target practice are inactive. During previous RFI activities conducted at SWMU 7, three new ranges were identified. These include: (1) West Trap Range; (2) East Trap Range; and (3) South Pistol Range. Since lead has not previously been investigated at SWMU 7, the ORR shooting lanes and target areas have been incorporated into this investigation. Samples will also be collected on the north facing hillside immediately south of the ORR target area and the South Pistol Range target area where impact may have occurred. Munitions handled at all of these locations consisted of small arms. During a preliminary site visit, no visual MEC was observed, and based on the nature of the site operation, MEC is not suspected to be present. MCs that might be present would

consist of lead shot fired from the weapons. Contamination would be located primarily in the surface soils including the drainage areas to the east of the site because lead mobility in soil is low. Due to the relatively close proximity of the sites making up UXO 7, the individual site features as noted below apply to UXO 7 as a whole. Site features and approximate boundaries for the individual sites that make up UXO 7 are presented on Figure 1-4.

1.4.2 Topography

UXO 7 comprises an irregularly shaped area in the southern to southwestern portion of the ORR. The area slopes gently to the east. Maximum ground surface elevations within UXO 7 are about 530 feet above msl, and the minimum elevation is about 500 feet above msl near NSWC Crane Highway 8 east of UXO 7, for a total relief of about 30 feet. The area east of UXO 7 continues to slope toward Turkey Creek (not shown on Figure 1-4), where the minimum elevation is approximately 485 feet above msl. The mean slope from the western side of UXO 7 to Turkey Creek is about 2 percent.

To the west and southwest of UXO 7, the ground surface rises steeply to a ridgetop that has a maximum elevation of about 710 feet above msl. Thus, the total relief from the top of the ridge to Turkey Creek is about 225 feet. A demolition range occupies the side slopes and top of the ridge to the west and southwest of UXO 7.

1.4.3 Geology

The geology is composed of residual soils formed by weathering of the underlying parent rock, unconsolidated alluvium, and Pennsylvanian- and Mississippian-aged sedimentary rock. Soil thickness ranges from less than 2 feet to a maximum of about 30 feet bgs. The northern portion of the site is covered with occasionally flooded Burnside loam, the western portion with Wellston silt loam, and the eastern portion with frequently flooded Haymond silt loam (McElrath, 1988). The variability of soil thickness at the ORR reflects the erosional effects of the ancestral Turkey Creek and its tributaries. The thickest soil corresponds to areas of deepest fluvial incision of the rock surface and subsequent alluvial (stream-deposited) and colluvial (slope debris) filling of the Turkey Creek Valley.

The sedimentary rock underlying the ORR is Mississippian-age sandstone from the Big Clifty Formation of the Mississippian-age Stephenson Group.

1.4.4 Soil and Vegetation Types

The vegetation at UXO 7 includes intermittently mowed grasslands, wooded slopes, and riparian wooded vegetation in the vicinity of the Turkey Creek drainage.

1.4.5 Hydrology

UXO 7 generally slopes toward Turkey Creek where the minimum elevation is approximately 485 feet above msl. Thus, a large portion of the site area drains directly into Turkey Creek. An unnamed perennial stream flows from northwest to southeast through the northern end of the ORR and drains into Turkey Creek. Thus, the northern portion of UXO 7 drains into the unnamed tributary prior to entering Turkey Creek. Another unnamed tributary creek flows from west to east through the southern end of UXO 7 and enters Turkey Creek. This tributary receives some surface water runoff from the southern end of UXO 7. It also drains some of the ridgetop area occupied by the Demolition Range (DR) (SWMU 6). A small man-made sediment retention basin (Pond 3) has been constructed on this unnamed tributary west of UXO 7. To aid in the settling of solid from Pond 3, an additional impoundment (Pond 3A) was constructed in the southeastern corner of the ORR (ponds are not shown on Figure 1-4).

All water and sediment discharging from UXO 7 eventually enters Turkey Creek.

1.4.6 Hydrogeology

The uppermost occurrence of groundwater is within the alluvium, which is present over much of the ORR. Groundwater elevations within the underlying Big Clifty-Beech Creek (BC/BC) aquifer are similar to those in the alluvium. The two units are hydraulically connected and considered to represent one unconfined aquifer in this area (Murphy and Wade, 1998). The occurrence and movement of groundwater is closely tied to the bedrock surface (USACE, 1991). Groundwater elevations within the site range from approximately 510 to 495 feet above msl, a difference of 15 feet. Flow direction is predominantly toward the east. Groundwater at the site is typically found between 10 to 20 feet bgs (TiNUS, 1999a).

The thick alluvial soils that occur over much of the area of UXO 7 constitute part of the uppermost aquifer. The similarity in water levels in wells installed solely in the soil column and wells installed in the upper Big Clifty Sandstone indicate that the soils are hydraulically connected with the BC/BC aquifer.

1.4.7 UXO 7 Conceptual Site Model

The CSM for UXO 7 was developed in a manner similar to the CSM for UXO 5 (see Section 1.3.7). The CSM is presented in a series of information profiles that present information about the site (see Table 1-2).

The configuration and past use of the site are such that MC releases to surface soil from the pistol range and trap shooting areas are expected to be of small quantities. Release of metals (lead) would occur through the deposition of lead shot dispersed over large areas. Thus, the majority of contamination is expected to exist in the top surfaces (upper 6 inches) of the surrounding soil. The contaminants may have migrated through surface soil to deeper soils and perhaps as deep as the groundwater table. However, the majority of contamination is expected to reside near or in surface soils because of the deposition mechanism and the very limited mobility of lead in soil. Surface soils pose the most significant exposure route for humans and ecological receptors.

1.4.8 UXO 7 Data Quality Objectives

The U.S. EPA DQO process was used to develop the sampling design for collecting UXO 7 RFA data. The seven steps of the process are described briefly below.

1.4.8.1 Step 1 - PROBLEM DEFINITION

The following is the problem definition:

1. MCs may be present in surface soils at the West Trap Range, the East Trap Range, the South Pistol Range, the ORR shooting lanes and target area, and the north facing hillside immediately south of both the South Pistol Range and the ORR.
2. If found to be present in surface soils, lead may have migrated deeper into subsurface soils, as well as groundwater, and any nearby sediment and surface water.
3. The CSM indicates that potentially complete pathways exist for human and ecological receptors both under current land uses and potential future land uses.
4. If present in significant concentrations in soils, lead could present risks to human and ecological receptors.

5. This RFA is being conducted to determine whether conditions exist that would warrant further study.

1.4.8.2 Step 2 - DECISIONS TO BE MADE

The primary goal of the RFA at UXO 7 is to obtain environmental data for use in making the following decisions:

1. Determine whether lead is present within any of the study areas in quantities or concentrations that require an immediate response. If so, initiate the appropriate response; otherwise, take no immediate action.
2. Determine whether lead is present within any of the study areas in quantities or concentrations that require proceeding to an RFI/Corrective Measures Study (RFI/CMS). If so, perform an initial risk evaluation; otherwise, do not investigate further.
3. If an initial risk evaluation indicates that a potentially unacceptable condition exists, proceed to an RFI, otherwise do not investigate further.

1.4.8.3 Step 3 - INPUTS REQUIRED TO MAKE THE DECISION

Data and information that will be required to make these decisions includes the following:

1. Lead concentrations in surface soils determined by SW-846 Method 3050B/6020 to assess whether lead is present in soils at concentrations greater than background and screening levels.
 - a. All UXO 7 surface soils belong to Soil Group 3 as defined in the NSWC Crane Basewide Soil Background Study (TtNUS, 2001). This knowledge will allow site and background soils to be matched so the data represent similar soils when conducting background comparisons.
 - b. Background lead data in soils similar to UXO 7 (from the NSWC Crane Basewide Soil Background Study). The 95% UTL from the background study will be used for comparisons.
2. Screening levels for lead to determine if it is necessary to proceed to expand the study area and whether or not to proceed to the RFI.
3. Method detection limits meeting screening levels.

4. The statistic for comparing lead concentrations to screening values.
5. A correlation coefficient between 0.65 and 1.0 is required when correlating field and laboratory lead concentrations. The correlation plot will be used to translate field concentrations to the equivalent fixed-base laboratory concentrations when the latter are not available for particular samples.

1.4.8.4 Step 4 - DELINEATION OF STUDY BOUNDARY

1. The horizontal boundary is defined as the area where lead shot is most likely to have been deposited as shown on Figure 1-4.
2. Lead, if present, would be primarily in surface soils with a maximum vertical depth of 2 feet.
3. Lateral expansion of the study area during an RFI phase may be necessary if lead is present at the lateral extent of the initial sampling pattern at concentrations greater than background (lead) and screening levels.
4. Vertical expansion of the study area during an RFI to subsurface soils may be necessary if lead is present in surface soils at concentrations greater than background (lead) and screening levels.
5. Vertical expansion of the study area during an RFI phase to groundwater may be necessary if MCs are present in surface soils at concentrations greater than background (metals) and screening levels.

1.4.8.5 Step 5 - DEFINITION OF RULES FOR DECISION MAKING

1. If lead is detected at concentrations in surface soils greater than background (lead) and screening values, perform an initial risk evaluation, otherwise investigate no further.
2. If lead is observed in any surface soil sample at concentrations greater than background concentrations and screening levels for lead at horizontal or vertical boundaries of sampling, expand the study area.
3. If initial risk evaluation shows that a potentially unacceptable condition exists, proceed to an RFI, otherwise investigate no further. The risk evaluation shall be based on laboratory or laboratory-equivalent lead concentrations.

1.4.8.6 Step 6 - PERFORMANCE CRITERIA

1. Because this is an RFA and depends heavily on biased sampling, probability limits for false decision errors were not established.
2. Performance criteria for new analytical data (lead only) are presented in the original QAPP.

1.4.8.7 PLAN FOR OBTAINING THE DATA

Sample Collection and Analyses

1. Surface Soil
 - Surface soil samples will be collected at 0 to 2 feet bgs in the areas shown on Figure 3-2.
 - Field (X-Ray Fluorescence) XRF analysis will be conducted to determine the locations for surface soil samples to be analyzed at a fixed-base laboratory.
 - Because the soil samples are shallow, they can be readily collected via a hand auger.
 - UXO clearance is not required because MEC has not been observed on site. However, the site workers will be vigilant for the presence of MEC. If MEC is observed, work shall cease and the Field Operations Leader shall be notified immediately so the presence of MEC can be communicated to the NSWC Crane Environmental Site Manager (ESM). Work will not continue until approved by this manager.
2. Analysis
 - Surface soils will be analyzed for lead (SW-846 3050B/6020).
3. Groundwater, surface water, and sediment samples have been collected and analyzed in previous SWMU 7/ORR investigations with results presented in the RFI Report. These media are not part of this investigation.

TABLE 1-1

**CONCEPTUAL SITE MODEL
UXO 5
NSWC CRANE DIVISION
CRANE, INDIANA
PAGE 1 OF 4**

Profile type	Information Needs	Preliminary Assessment findings
Range/Site Profile	Installation Name	NSWC Crane
	Installation Location	Crane, Martin County, Indiana
	Range/Site Name	B-2044 Drop Tower/Test Rail
	Range/Site Location	Central portion of NSWC Crane Division
	Range/Site History	Used for the drop testing of 20-mm ammunition and ejection seat testing using CADs/PADs on the test rail. Site was first used January 1951 and remained in use until December 1973.
	Range/Site Area and Layout	Approximately 0.013 acres
	Range/Site Structures	A 100-ft. high metal drop tower exists at the site, as well as a metal test rail and a small open building used as a preparation area.
	Range/Site Boundaries	N: Highway 304A S: Wooded Area E: Wooded Area W: Highway 45
	Range/Site Security	The installation is fenced. Once inside installation fence-line, there are no other barriers to the site.
Munitions/Release Profile	Munitions types	20-mm cartridges, CADs/PADs
	Maximum probability Penetration Depth	Surface only
	MEC Density	Minimal range related debris is expected to be at the site based upon the Navy operating procedures to collect and remove related debris immediately after testing. These are known or suspected MEC areas.
	MEC Scrap/Fragments	None found during site visit.
	Associated Munitions Constituents	Minimal amount of meals and other inorganics.
	Migration Routes/Release Mechanisms	If MC were present at the site, it would only be located within surface soils (less than 2 feet bgs). MC are not anticipated to be present at the B-2044 Drop Tower/Test Rail; however, if MC are present at the site, they may migrate via soil and groundwater.

TABLE 1-1
CONCEPTUAL SITE MODEL
UXO 5
NSWC CRANE DIVISION
CRANE, INDIANA
PAGE 2 OF 4

Profile type	Information Needs	Preliminary Assessment findings
Physical Profile	Climate	Temperature climate zone, which has a wide temperature range between summer and winter
	Topography	Flat to gently undulating terrain
	Geology	The area is located on the eastern flank of the Illinois Basin consisting of shale, sandstone, limestone, and coal beds.
	Soil	ZnB-Zanesville-Udorthents Complex, 6 to 12% slopes
	Hydrogeology	No wells located on site. Groundwater is not used for drinking.
	Hydrology	NSWC Crane is located within the Lower East Fork White watershed. The East Fork White River flows approximately 40 miles southwest before joining the Muscatatuck River which eventually joins the Ohio River. NSWC Crane is located approximately 10 miles northwest of the East Fork White River.
	Vegetation	The area is covered with low grass and is surrounded by wooded land.
Land Use and Exposure Profile	Current land Use	The site is no longer used for testing and is considered closed. There is no anticipated plan to reuse the site in the future for testing purposes. The land surrounding the drop tower and test rail is forested and is covered with heavy grass and vegetation. There are no anticipated uses for the site.
	Current Human Receptors	Naval personnel, contractors, and authorized visitors.
	Current Activities (frequency, nature of activity)	No activity currently at the site.
	Potential Future land Use	No change in land use is expected.
	Potential Future Human Receptors	Naval personnel, contractors, and authorized visitors.
	Potential Future Land Use-Related Activities:	No change in land use is expected.
	Zoning/Land Use Restrictions	No known zoning/land use restrictions

TABLE 1-1
CONCEPTUAL SITE MODEL
UXO 5
NSWC CRANE DIVISION
CRANE, INDIANA
PAGE 3 OF 4

Profile type	Information Needs	Preliminary Assessment findings
Land Use and Exposure Profile (Continued)	Demographics/Zoning	NSWC Crane employs 4,000 personnel, both civilian and military. Martin County has a population of 10,383, which comprises 0.16 percent of the state's population. The population per square mile in Martin County is 30.9.
	Beneficial Resources	The NSWC Crane forest has been important to the re-establishment of deer, turkey, ruffed grouse, and eagles in Indiana. Timber is also harvested at NSWC Crane.
Ecological Profile	Habitat Type	Grassland and woodlands
	Degree of Disturbance	If current conditions continue, the degree of disturbance at the site will be low. Low indicates that the site is/will be unused, and habitat for species present are/will be undisturbed (i.e., undisturbed grasslands, woodlands). Periodic activities will occur such as mowing and building maintenance.
	Ecological Receptors	
	Federal Endangered Species:	No federal endangered species are known at the site.
	Federal Threatened Species:	The Bald Eagle (<i>Haliaeetus leucocephalus</i>) could be located at the site based on the large hunting range of the eagle.
	State Endangered Species:	The potential for the Grasshopper sparrow, bobcat, osprey, timber rattlesnake, and yellow-crowned night heron exists; however, none have been identified at the site.
	State Threatened Species:	The Bald Eagle (<i>Haliaeetus leucocephalus</i>) could be located at the site based on the large hunting range of the eagle.
	Other Ecological Receptors:	The habitat at the site and the surrounding area is home to many woodland wildlife species, such as deer, rabbits, raccoons, and wild turkeys. There is the potential for wildlife to burrow, forage, and nest on the site.

TABLE 1-1
CONCEPTUAL SITE MODEL
UXO 5
NSWC CRANE DIVISION
CRANE, INDIANA
PAGE 4 OF 4

Profile type	Information Needs	Preliminary Assessment findings
Ecological Profile (Continued)	Relationship of MEC/MC Sources to Habitat and Potential Receptors	Ecological receptors may come into direct contact with MEC/MC (in the soil or on the surface). Wildlife that inhabit or utilize the area may come into contact with MC that has been incorporated into the food chain (bio-accumulated in plants and animals). Ecological receptors contacting MEC and creating an explosive hazard is not likely but should be considered.

TABLE 1-2

**CONCEPTUAL SITE MODEL
UXO 7
NSWC CRANE DIVISION
CRANE, INDIANA
PAGE 1 OF 4**

Profile Type	Information Needs	Preliminary Assessment Findings
Range/Site Profile	Installation Name	NSWC Crane.
	Installation Location	Crane, Martin County, Indiana.
	Range/Site Name	Comprised of several individual locations: (1) West Trap Range; (2) East Trap Range; (3) South Pistol Range; (4) ORR shooting lanes and target area.
	Range/Site Location	Central portion of NSWC Crane Division.
	Range/Site History	Used for target practice and possible trap shooting utilizing small arms (shotguns, rifles, pistols).
	Range/Site Area and Layout	Located within the ORR which comprises approximately 20 acres of SWMU 7.
	Range/Site Structures	Open grassy areas with several earth berms for shooting backgrounds.
	Range/Site Boundaries	N: Open area of northern section of the ORR. S: Wooded E: Highway 8/wooded W: Wooded/DR
	Range/Site Security	There are no physical barriers to site access.
Munitions/Release Profile	Munitions Types	Small arms.
	Maximum Probability Penetration Depth	Surface only (less than 6 inches bgs).
	MEC Density	Minimal range-related debris is expected to be at the site based on the Navy operating procedures of collecting and removing related debris immediately after firing and based on the dispersion pattern of shot.
	MEC Scrap/Fragments	None found during site visit.
	Associated Munitions Constituents	Minimal amount of metals (lead) from the shot. Other contaminants may be present, but lead shot is expected to dominate.

TABLE 1-2

**CONCEPTUAL SITE MODEL
UXO 7
NSWC CRANE DIVISION
CRANE, INDIANA
PAGE 2 OF 4**

Profile Type	Information Needs	Preliminary Assessment Findings
Munitions/Release Profile (Continued)	Migration Routes/Release Mechanisms	If MCs were present at the site, they are presumed to be located within surface soils (less than 2 feet bgs). MCs are not anticipated to be present at any of the three shooting ranges; however, if MCs are present at the site, they may migrate via soil and groundwater. Overland flow could carry contaminated surface soil to downgradient locations and leaching by precipitation could move contaminants deeper into soils and eventually to groundwater.
Physical Profile	Climate	Temperate climate zone that has a wide temperature range between summer and winter.
	Topography	Flat to gently undulating terrain.
	Geology	The area is located on the eastern flank of the Illinois Basin consisting of shale, sandstone, limestone, and coal beds.
	Soil	Residual soils formed by weathering of the underlying parent rock, unconsolidated alluvium, and Pennsylvanian- and Mississippian-aged rock. Soil thickness ranges from 2 feet to a maximum of 30 feet bgs.
	Hydrogeology	Permanent groundwater monitoring wells located on site. Groundwater is not used for drinking.
	Hydrology	NSWC Crane is located within the Lower East Fork White River watershed. Drainage channels convey UXO 7 surface flow and, where elevations facilitate, groundwater flow, to lower elevations. This flows to the East Fork White River located approximately 40 miles southwest before joining the Muscatatuck River, which eventually joins the Ohio River. NSWC Crane is located approximately 10 miles northwest of the East Fork White River.
	Vegetation	The area is covered with low grass and is surrounded by wooded land.

TABLE 1-2

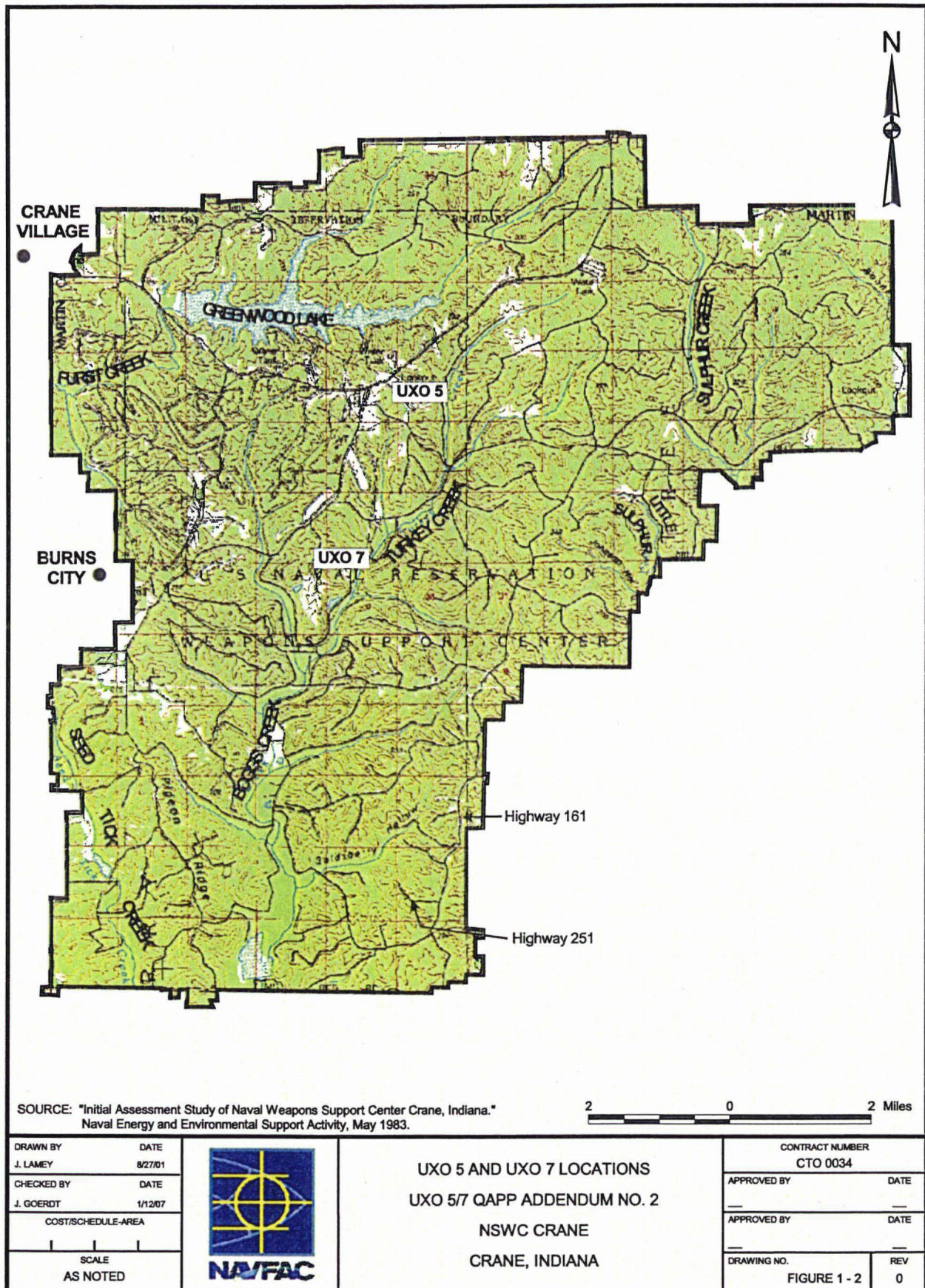
CONCEPTUAL SITE MODEL
UXO 7
NSWC CRANE DIVISION
CRANE, INDIANA
PAGE 3 OF 4

Profile Type	Information Needs	Preliminary Assessment Findings
Land Use and Exposure Profile	Current Land Use	The site is no longer used as a pistol range or for trap shooting. There is no plan to reuse the site in the future for a range or trap shoot site. The land surrounding the pistol range and trap shooting areas is forested and is covered with heavy grass and vegetation. There are no anticipated uses for the site in the foreseeable future.
	Current Human Receptors	Naval personnel, contractors, and authorized visitors.
	Current Activities (frequency, nature of activity)	No activity currently at the site.
	Potential Future Land Use	No change in land use is expected in the foreseeable future.
	Potential Future Human Receptors	Naval personnel, contractors, and authorized visitors.
	Potential Future Land Use-Related Activities	No change in land use is expected in the foreseeable future.
	Zoning/Land Use Restrictions	No known zoning/land use restrictions.
	Demographics/Zoning	NSWC Crane employs 4,000 personnel, both civilian and military. Martin County has a population of 10,383, which comprises 0.16 percent of the State's population. The population per square mile in Martin County is 30.9.
	Beneficial Resources	The NSWC Crane forest has been important to the re-establishment of deer, turkey, ruffed grouse, and eagles in Indiana. Timber is also harvested at NSWC Crane.
Ecological Profile	Habitat Type	Grassland and woodlands.
	Degree of Disturbance	Under current conditions, the degree of disturbance at the site is low. Low indicates that the site is/will be unused, and habitat for species present are/will be undisturbed (i.e., undisturbed grasslands, woodlands). Periodic activities will occur such as mowing and building maintenance.
	Federal Endangered Species	No federal endangered species are known at the site.


TABLE 1-2
CONCEPTUAL SITE MODEL
UXO 7
NSWC CRANE DIVISION
CRANE, INDIANA
PAGE 4 OF 4

Profile Type	Information Needs	Preliminary Assessment Findings
Ecological Profile (Continued)	Federal Threatened Species	The Bald Eagle (<i>Haliaeetus leucocephalus</i>) could be located at the site based on the large hunting range of the eagle.
	State Endangered Species	The potential for the Grasshopper sparrow, bobcat, osprey, timber rattlesnake, and yellow-crowned night heron exists; however, none have been identified at the site.
	State Threatened Species	The Bald Eagle (<i>Haliaeetus leucocephalus</i>) could be located at the site based on the large hunting range of the eagle.
	Other Ecological Receptors	The habitat at the site and the surrounding area is home to many woodland wildlife species such as deer, rabbits, raccoons, and wild turkeys. There is the potential for wildlife to burrow, forage, and nest on the site.
	Relationship of MEC/MC Sources to Habitat and Potential Receptors	Ecological receptors may come into direct contact with MEC/MCs (in the soil or on the surface). Wildlife that inhabit or utilize the area may come into contact with MCs that have been incorporated into the food chain (bio-accumulated in plants and animals). Ecological receptors contacting MEC and creating an explosive hazard are not likely but should be considered.







DRAWN BY S. STROZ		DATE 2/28/07			CONTRACT NUMBER 0034					
CHECKED BY J. GOERDT		DATE 3/7/07			APPROVED BY _____		DATE _____			
COST/SCHEDULE-AREA 						APPROVED BY _____		DATE _____		
SCALE AS NOTED						DRAWING NO. FIGURE 1 - 4			REV 0	

UXO 7

2003 AERIAL PHOTOGRAPHY

UXO 5/UXO 7 QAPP ADDENDUM NO. 2

NSWC CRANE

CRANE, INDIANA

200 0 200 Feet

2.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

This section describes the project management, organization, and responsibilities of key personnel for the RFAs described in Section 1.0 at NSWC Crane UXO 5 and UXO 7. The project organizational structures are presented below for both UXO sites.

TtNUS, on behalf of the United States Navy, is responsible for the preparation for this document and for oversight of the site investigations to ensure that they are completed satisfactorily at each of the two UXO sites.

The subcontracted laboratory for each UXO site is responsible for analyzing all confirmation samples in accordance with the analytical methods and additional requirements specified in this document. It also will be the responsibility of the analytical laboratory to properly dispose of unused sample aliquots.

2.1 MANAGEMENT

Individual key persons responsible for work to be conducted under this QAPP Addendum No. 2 are identified on Figures 2-1 and 2-2. Corresponding addresses and telephone numbers of key personnel are listed by organization in Table 2-1. Indiana Department of Environmental Management (IDEM), U.S. EPA, Navy, and TtNUS personnel are identified in the corresponding table and figure.

TtNUS personnel will report directly to NAVFAC SE. TtNUS will maintain open channels of communication during field activities to facilitate resolution of problems as efficiently as possible. Personnel from the Navy will be actively involved and will coordinate with TtNUS personnel as necessary. Responsibilities for program management, project management, field operations, and laboratory operations are discussed in the following sections, as appropriate. It is intended that the individuals named will assume the designated responsibilities to the extent that the specific person is available to perform the stated activities.

2.1.1 Indiana Department of Environmental Management Corrective Action Project Manager

The IDEM Corrective Action Project Manager (PM), Mr. Doug Griffin, will oversee the implementation of the RFA at UXO 5. The IDEM PM represents IDEM's interests and will provide input from this perspective and lend general technical assistance to NSWC Crane field activities.

2.1.2 U.S. EPA Oversight

The U.S. EPA Region 5 PM, Peter Ramanauskas, will oversee the implementation of the RFA at UXO 7. He will also represent U.S. EPA's interests and will provide input from this perspective and lend general historical and technical assistance to NSWC Crane field activities.

2.1.3 Navy Project Manager

The Navy Remedial Project Manager (RPM), Mr. Howard Hickey will act as the focal representative for the United States Navy, providing management, technical direction, and oversight for all NSWC Crane project activities performed by contractors (e.g., TtNUS) and their subcontractors. In matters such as facilitation of site access, oversight, etc., the Navy RPM is assisted by the NSWC Crane Environmental Site Manager (ESM), Mr. Tom Brent.

2.1.4 TtNUS Program and Project Management

TtNUS Program Manager

The TtNUS NAVFAC SE CLEAN Program Manager (ProgMan), Ms. Debra Humbert, provides operations, technical, and administrative leadership and oversees and supports quality policies. The ProgMan assigns project Task Order Managers (TOMs) and oversees their performance. The ProgMan also ensures the availability of technical and support resources for program operations, and maintains consistency in procedures and projects among CTO assignments. In these matters, the ProgMan is assisted by the TOMs.

TtNUS Task Order Manager

The TtNUS TOM, Mr. Ralph Basinski, has overall responsibility for ensuring that the project meets U.S. EPA and IDEM objectives and Navy and TtNUS quality standards associated with investigation oversight, sample collection, sample analyses, and data interpretation to determine whether the RFAs are completed satisfactorily. The TOM is responsible for the preparation and distribution of this QAPP Addendum No. 2. The TOM will report to the Navy RPM and is responsible for technical QC and project oversight. Additional responsibilities of the TOM are as follows:

- Ensuring timely resolution of project-related technical, quality, and safety questions associated with TtNUS operations.
- Functioning as the primary TtNUS interface with the Navy RPM, NSWC Crane ESM, TtNUS field and office personnel, and laboratory points of contact.
- Ensuring that TtNUS health and safety issues related to this project are communicated effectively to all personnel and off-site laboratories.
- Monitoring and evaluating all TtNUS subcontractor performance.
- Coordinating and overseeing work performed by TtNUS field and office technical staff (including data validation, data interpretation, and report preparation).
- Coordinating and overseeing maintenance of all TtNUS project records.
- Coordinating and overseeing review of TtNUS project deliverables.
- Preparing and issuing final TtNUS deliverables to the Navy.

TtNUS Project Chemist

The TtNUS Project Chemist, Mr. Edward Sedlmyer, has overall responsibility for ensuring that the project meets objectives from the standpoint of laboratory performance. The Project Chemist is responsible for the technical preparation of laboratory statements of work (SOWs) and work releases. The PM of the subcontracted laboratory conducting confirmation sample analyses will report to the TtNUS Project Chemist. The Project Chemist will report to the TOM. Additional responsibilities of the Project Chemist are as follows:

- Providing technical advice to the TtNUS team on matters of project chemistry.
- Monitoring and evaluating subcontractor laboratory performance.
- Ensuring timely resolution of laboratory-related technical, quality, or other issues effecting project goals.
- Functioning as the primary interface with the subcontracted laboratory and the TOM.
- Coordinating and overseeing work performed by the subcontracted laboratory.

- Coordinating and overseeing review of laboratory deliverables.
- Recommending appropriate laboratory corrective actions.

TtNUS Health and Safety Manager

The TtNUS Health and Safety Manager (HSM), Mr. Matt Soltis, is responsible for the following:

- Providing technical advice to the TOM on matters of health and safety.
- Overseeing the development and review of the Health and Safety Plan (HASP).
- Conducting health and safety audits.
- Preparing health and safety reports for management.

Each field event will have the support of a health and safety officer who will be assigned by the HSM.

2.2 QUALITY ASSURANCE

This section identifies the QA responsibilities for the UXO RFAs. Responsibilities of U.S. EPA, IDEM, TtNUS, and the analytical laboratory are discussed.

2.2.1 Indiana Department of Environmental Management and U.S. EPA

The IDEM chemist, Mr. Craig Pender, and U.S. EPA chemist, Mr. Allen Debus, are responsible for review and approval of chemistry aspects of this QAPP Addendum No. 2 and for providing overall QA support and review. Additional responsibilities may include the following:

- Coordinating external performance and system audits of the contracted laboratory
- Reviewing and evaluating analytical field and laboratory procedures

2.2.2 TtNUS QA Coordinator

The TtNUS NAVFAC SE Program Quality Assurance Manager (QAM), Mr. Richard Ninesteel, is responsible for overall QA for the TtNUS portion of the project. He reports directly to the TtNUS ProgMan. He acts on behalf of the United States Navy for project QA. The Quality Assurance Manager (QAM) is responsible for the following:

- Developing, maintaining, and monitoring QA policies and procedures.
- Providing training to TtNUS staff in QA/QC policies and procedures.
- Conducting systems and performance audits to monitor compliance with environmental regulations, contractual requirements, QAPP requirements, and corporate policies and procedures.
- Auditing project records.
- Monitoring subcontractor quality controls and records.
- Assisting in the development of corrective action plans and ensuring correction of nonconformances reported in internal or external audits.
- Ensuring that this QAPP Addendum meets TtNUS, Navy, and IDEM requirements.
- Overseeing the responsibilities of the TtNUS Project QA/QC Advisor.
- Preparing QA reports for management.

2.2.3 TtNUS Project QA Advisor

The TtNUS Project QA Advisor, Dr. Tom Johnston, supports the TOM in preparing and reviewing the QAPP and in conducting data assessments. The Project QA Advisor communicates directly with the QAM on matters of QA/QC.

2.2.4 Laboratory Responsibilities

The subcontracted laboratory for the RFAs is responsible for analyzing all samples in accordance with the analytical methods and additional requirements specified in this QAPP Addendum No. 2. It will also be the analytical laboratory's responsibility to properly dispose of unused sample aliquots. Responsibilities of key laboratory personnel are outlined in the following paragraphs.

Laboratory Project Managers

The Laboratory PM will interface directly with the TtNUS Project Chemist, TtNUS TOM, and TtNUS QA Advisor and will perform the following tasks:

- Ensure that methods and project-specific requirements are properly communicated and understood by laboratory personnel.
- Ensure that all laboratory resources are available on an as-required basis.
- Ensure compliance with analytical and project QA requirements.
- Review data packages for completeness, clarity, and compliance with project requirements.
- Inform the TtNUS TOM of project status and any sample receipt or analytical problems.
- Oversee the preparation of and approving final analytical reports before submittal to TtNUS.

Laboratory Operations Manager

Responsibilities of the Laboratory Operations Manager include the following:

- Supporting the QA program within the laboratory
- Providing management overview of both production- and quality-related laboratory activities
- Maintaining adequate staffing to meet project analytical and quality objectives
- Approving all laboratory SOPs and QA documents
- Supervising in-house chain-of-custody documentation

Laboratory Quality Assurance Officer

The Laboratory QA Officer (QAO) will report directly to the Laboratory Operations Manager and will be independent of laboratory production management to ensure that laboratory quality performance is assessed without schedule and cost considerations. Responsibilities of the Laboratory QAO include the following:

- Defining appropriate laboratory QA procedures and monitoring overall laboratory QA.
- Stopping work if a condition adverse to the quality of work is encountered, if QA or QC procedures are not followed, or if analytical out-of-control events are encountered that have not been corrected.
- Approving and maintaining document control of all QA documents and SOPs.

- Performing and/or implementing internal system and performance audits and verifying completion of corrective actions cited in audits.
- Directing laboratory participation in laboratory accreditation and certification programs.

Laboratory Sample Custodian

The Laboratory Sample Custodian will report to the Laboratory Operations Manager. Responsibilities of the Laboratory Sample Custodian include the following:

- Receiving and inspecting the incoming sample containers.
- Recording conditions of incoming sample containers.
- Signing appropriate documents.
- Verifying chain-of-custody records.
- Notifying the Laboratory PM of sample receipt and inspection.
- Assigning a unique identification number and customer number and entering each into the sample receiving log.
- With the help of the Laboratory PM, initiating transfer of the samples to appropriate laboratory sections.
- Controlling and monitoring access/storage of samples and extracts.

Laboratory Technical Staff

The laboratory technical staff will be responsible for sample analyses based on the analytical methods and requirements specified in this QAPP Addendum No. 2.

2.3 FIELD INVESTIGATION

TtNUS will be responsible for all field sampling and analysis activities related to confirming that the RFAs are completed satisfactorily. The TtNUS field team will be organized according to the activities planned. Field team members will be selected based on the type and extent of effort required. All team members will be appropriately skilled and trained for the tasks they are assigned to perform

2.3.1 Field Operations Leader

The TtNUS FOL is responsible for coordinating all on-site personnel and for providing technical assistance, when required. The FOL has authority to complete the field work in the most efficient manner while adhering to the requirements of this QAPP Addendum No. 2 and the HASP. The TtNUS FOL will ensure the availability and maintenance of all sampling materials and equipment pertinent to his/her function. The TtNUS FOL is responsible for completing all sampling, field, and chain-of-custody documentation, will assume custody of all samples sent to the confirmation sampling laboratory, and will ensure the proper handling and shipping of samples. The TtNUS FOL will report directly to the TtNUS TOM but will have authority to guide field operations to efficiently conclude the scheduled work. Specific TtNUS FOL responsibilities include the following:

- Ensuring that all health and safety requirements unique to these RFAs are implemented.
- Functioning as the on-site communications link between field staff members, the Site Safety Officer (SSO), NSWC Crane ESM, and TtNUS TOM.
- Alerting off-site analytical laboratories of any special health and safety hazards associated with environmental samples.
- Overseeing the mobilization and demobilization of all field equipment and subcontractors.
- Coordinating and managing the field technical staff.
- Adhering to the work schedules provided by the TtNUS TOM.
- Ensuring the proper maintenance of site logbooks, field logbooks, and field recordkeeping.

- Initiating field task modification requests (field change orders) when necessary.
- Identifying and resolving problems in the field, resolving difficulties via consultation with the NSW Crane ESM, implementing and documenting corrective action procedures, and providing communication between the field team and project management.

2.3.2 Site QA/QC Advisor

The FOL (or designee) will act as the Site QA/QC Advisor and will be responsible for ensuring adherence to all QA/QC requirements as defined in this QAPP Addendum No. 2. Strict adherence to these procedures is critical to the collection of acceptable and representative data. The following is a summary of the Site QA/QC Advisor's responsibilities:

- Ensuring that field QC samples are collected at the proper frequencies.
- Ensuring that additional volumes of sample are supplied to the analytical laboratory with the proper frequency to accommodate laboratory QA/QC analyses.
- Ensuring that measuring and test equipment are calibrated, used, and maintained in accordance with applicable procedures and technical standards.
- Acting as liaison between site personnel, laboratory personnel, and the QAM.
- Managing bottleware shipments and overseeing field preservation.

2.3.3 Site Safety Officer

The duties of the designated TtNUS SSO are detailed in the TtNUS HASP. The SSO has stop-work authority, which can be executed upon the determination of an imminent safety hazard.

2.3.4 Technical Staff

The technical staff for this project will be drawn from a pool of qualified personnel. All of the designated team members will be experienced professionals who possess the degree of specialization and technical competence required to effectively and efficiently perform the required work.

Field staff members are responsible for complying with field-related requirements as presented in this QAPP Addendum No. 2 and the HASP.

2.4 SPECIAL TRAINING REQUIREMENTS AND CERTIFICATIONS

All field personnel will have appropriate training to conduct the field activities to which they are assigned. Additionally, each site worker will be required to have completed a 40-hour course (and 8-hour refresher course, if applicable) in Health and Safety Training as described under Occupational Safety and Health Administration (OSHA), 29 Code of Federal Regulations (CFR) 1910.120(b)(4).

Users of the XRF instrument must be properly trained to correctly operate the instrument by a certified user of the instrument or by the instrument manufacturer.

TABLE 2-1

**KEY PROJECT PERSONNEL FOR UXO 5 AND UXO 7
NAMES, PHONE NUMBERS, AND ADDRESSES
UXO 5 AND UXO 7 QAPP ADDENDUM NO. 2**

**NSWC CRANE
CRANE, INDIANA
PAGE 1 OF 2**

PERSON / TITLE / ORGANIZATION	ADDRESS	TELEPHONE
Peter Ramanauskas U.S. EPA Oversight U.S. EPA Region 5	EPA Region 5 77 West Jackson Blvd. Chicago, Illinois 60604	Phone: (312) 886-7890 FAX: (312) 353-4788
Howard Hickey Remedial Project Manager U.S. Navy NAVFAC	Department of the Navy Building 1A, Code 45313 201 Decatur Avenue Great Lakes, IL 60080	Phone: (847) 688-5999 FAX: (847) 688-2319
Doug Griffin Corrective Action Project Manager Office of Land Quality Hazardous Waste Permits IDEM	Corrective Action Section Office of Land Quality Hazardous Waste Permits 100 N. Senate Avenue P. O. Box 6015 Indianapolis, Indiana 46206-6015	Phone: (313) 233-2710
Craig Pender Project Chemist Office of Land Quality IDEM	Office of Land Quality Hazardous Waste Permits 100 N. Senate Avenue P. O. Box 6015 Indianapolis, Indiana 46206-6015	Phone: (313) 234-0941
Tom Brent Environmental Manager NSWC Crane	NSWC Crane Code PRCR4-TB B-3260 300 Highway 361 Crane, Indiana 47522-5009	Phone: (812) 854-6160 FAX: (812) 854-4177
Debra Humbert Program Manager Tetra Tech NUS	Tetra Tech NUS 661 Andersen Drive Pittsburgh, Pennsylvania 15220-2745	Phone: (412) 921-8968 FAX: (412) 921-4040
Richard Ninesteel Quality Assurance Manager Tetra Tech NUS	Tetra Tech NUS 661 Andersen Drive Pittsburgh, Pennsylvania 15220-2745	Phone: (412) 921-8746 FAX: (412) 921-4040
Matt Soltis Health and Safety Manager Tetra Tech NUS	Tetra Tech NUS 661 Andersen Drive Pittsburgh, Pennsylvania 15220-2745	Phone: (412) 921-8912 FAX: (412) 921-4040
Ralph Basinski Task Order Manager Tetra Tech NUS	Tetra Tech NUS 661 Andersen Drive Pittsburgh, Pennsylvania 15220-2745	Phone: (412) 921-8308 FAX: (412) 921-4040
James Goerdts Field Operations Leader Tetra Tech NUS	Tetra Tech NUS 661 Andersen Drive Pittsburgh, Pennsylvania 15220-2745	Phone: (412) 921-8425 FAX: (412) 921-4040

TABLE 2-1

**KEY PROJECT PERSONNEL FOR UXO 5 AND UXO 7
NAMES, PHONE NUMBERS, AND ADDRESSES
UXO 5 AND UXO 7 QAPP ADDENDUM NO. 2
NSWC CRANE
CRANE, INDIANA
PAGE 2 OF 2**

PERSON / TITLE / ORGANIZATION	ADDRESS	TELEPHONE
Joseph Samchuck Data Validation Manager Tetra Tech NUS	Tetra Tech NUS 661 Andersen Drive Pittsburgh, Pennsylvania 15220-2745	Phone: (412) 921-8510 FAX: (412) 921-4040
Edward Sedlmyer Project Chemist Tetra Tech NUS	Tetra Tech NUS 661 Andersen Drive Pittsburgh, Pennsylvania 15220-2745	Phone: (412) 921-8704 FAX: (412) 921-4040
Tom Johnston Quality Assurance Advisor Tetra Tech NUS	Tetra Tech NUS 661 Andersen Drive Pittsburgh, Pennsylvania 15220-2745	Phone: (412) 921-8615 FAX: (412) 921-4040
Ann Ho Project Manager Laucks Testing Laboratories, Inc.	Laucks Testing Laboratories, Inc. 940 South Harney Street Seattle, WA 98108	Phone: (206) 957-2428 FAX: (206) 767-5063
Harry Romberg Laboratory Quality Assurance Manager Laucks Testing Laboratories, Inc.	Laucks Testing Laboratories, Inc. 940 South Harney Street Seattle, WA 98108	Phone: (206) 767-5060 FAX: (206) 767-5063

FIGURE 2-1

PROJECT ORGANIZATION CHART FOR UXO 5
UXO 5 AND UXO 7 QAPP ADDENDUM NO. 2
NSWC CRANE
CRANE, INDIANA

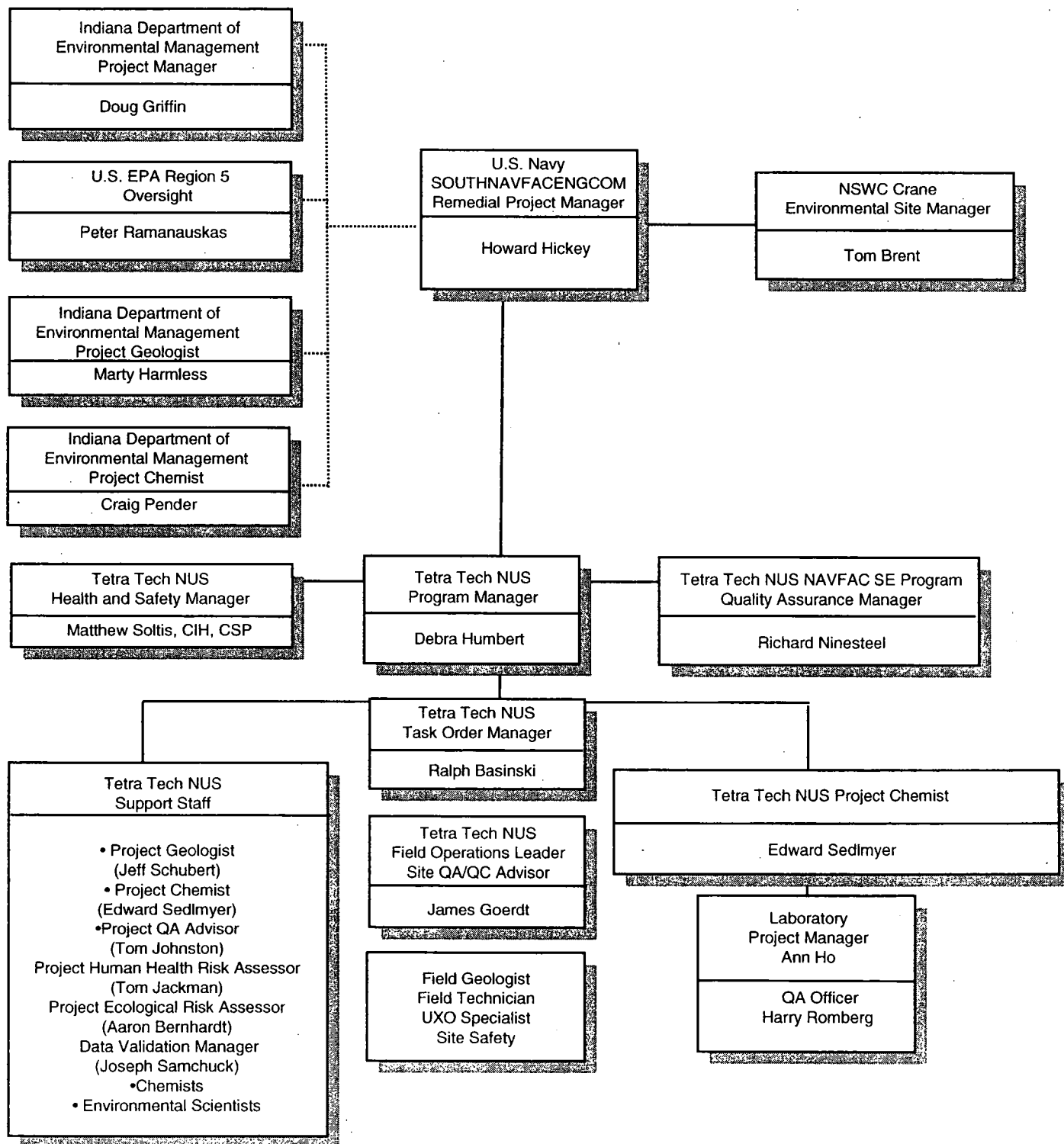
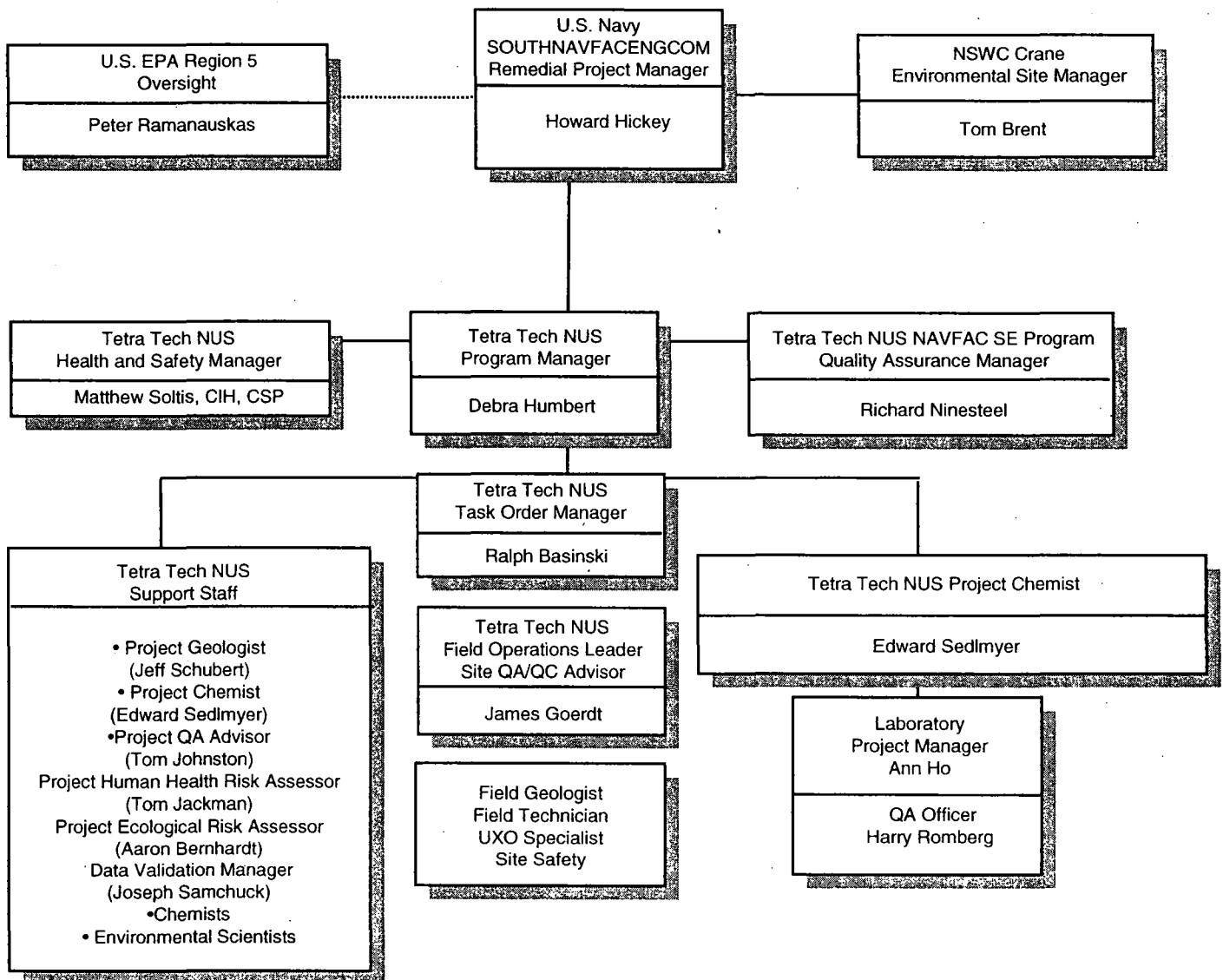


FIGURE 2-2

PROJECT ORGANIZATION CHART FOR UXO 7
UXO 5 AND UXO 7 QAPP ADDENDUM NO. 2
NSWC CRANE
CRANE, INDIANA



3.0 SAMPLING AND ANALYSIS PLAN

3.1 GENERAL APPROACH

This section describes sampling locations, methods, and rationales for the sampling activities to be conducted in support of the RFAs at UXO 5 and UXO 7. The equipment and procedures to be used for collecting, handling, preserving, recording, logging, and shipping samples to an analytical laboratory are presented in this QAPP Addendum No. 2. Electronic data collection may be used during these RFAs. Field SOPs for use by field personnel can be found in Appendix A, and laboratory SOPs are presented on CD in Appendix B. The existing HASP used for the RFI at SWMU 7 (which encompasses UXO 7) has been updated and modified to include the additional work at UXO 5. All surface soil sampling will be performed using hand augers.

Prior to any field activities, the TOM will ensure that all field personnel read and understand this QAPP Addendum No. 2 and the associated HASP, the FOL will ensure that all required field equipment for non-health-and-safety-related equipment is available and operational, and the SSO will ensure that all health-and-safety-related equipment is available and operational.

Surface soil sampling activities at the following sites are described in this SAP:

- UXO 5 – Building 2044 Drop Tower and Test Rail
- UXO 7 – West Trap Range, East Trap Range, South Pistol Range, ORR shooting lanes and target area.

Boring activities, sampling, and site characterization work at these two UXO sites are described in this section.

All field visual observations including physical observation of lead shot will be recorded on field log sheets. Any encounters with metallic objects, or other objects that indicate a potential contaminant source or hazard, will be reported to the FOL and the SSO, and appropriate actions will be taken as specified in this QAPP Addendum No. 2 and the HASP.

3.2 SAMPLE IDENTIFICATION SYSTEM

All samples collected for fixed-base laboratory analyses will be properly labeled with an adhesive-backed sample label affixed to each sample container in accordance with SOP CTO034-01 (Appendix A).

The surface soil samples at UXO 5 will be composite samples from multiple aliquots (discrete samples) collected from a grid representing specific areas within UXO 5. The aliquots will be collected from the 0-2 foot bgs interval. Composite samples will be formed by blending appropriate aliquots together (as described in Section 3.3.1), and the composite samples will be labeled in accordance with SOP CTO034-02 (Appendix A). Individual aliquots will be designated as "A," "B," "C," and "D," to indicate sequential aliquots collected for compositing. The locations where the aliquots are acquired will be noted in the field logbook.

The surface soil samples at UXO 7 will be grab samples collected from a grid system representing specific locations within each of the investigation areas.

Each collected sample will be assigned a unique sample tracking number as described in SOP CTO034-02 (Appendix A). Any other pertinent information regarding sample identification will be recorded in the field logbooks and on sample log sheets.

The sampling time recorded on the Chain-of Custody form and labels for duplicate samples will be 0000 so that the duplicate samples are "blind" to the laboratory. Notes detailing the sample number, time, date, and type will be recorded on the sample log sheets and will document the location of the duplicate sample (sample log sheets are not provided to the laboratory).

Matrix spike (MS) and matrix spike duplicate (MSD) samples will be designated on the field documentation and Chain-of-Custody forms (see SOP CTO034-03).

3.3 SAMPLING LOCATIONS, ANALYSES, AND RATIONALES

This section identifies sampling locations, QA/QC samples to be collected, analyses to be performed, and rationales for the sampling and analytical program. Details regarding the preservation, packaging, and shipping of samples are included in Section 3.6. For both UXO 5 and UXO 7, all samples collected from the surface soil interval (0 to 2 feet deep) will be prepared and analyzed according to the normal laboratory protocol. Soil sampling locations are shown on Figures 3-1 (UXO 5) and 3-2 (UXO 7).

If MEC is observed in any boring sample or near any work area, work must be halted. The presence of MEC must be communicated to the FOL, and the FOL will then communicate with the NSWC Crane EDM so the appropriate action may be taken. If obvious soil staining is observed in any boring samples, the staining will be described in the boring log, and additional samples may be collected at the discretion of

the FOL or site geologist to determine the nature and possibly the extent of associated site-related contamination.

The total numbers of soil analyses for each analyte group are tabulated in Table 3-1 for UXO 5 and UXO 7. Soil QA/QC samples will be collected at frequencies listed in Table 3-2 for UXO 5 and UXO 7. Table 3-3 presents a summary of the sample analyses, container types and volumes, preservation requirements, and holding times for the samples to be collected at UXO 5 and UXO 7.

3.3.1 Sampling Strategy

All surface soil sampling will be performed using hand auger soil sampling techniques. Details regarding soil sampling equipment and procedures are included in Section 3.4 and SOP CTO034-04.

The sampling designs for UXO 5 and UXO 7 are stratified with the intent to sample most densely those areas that are likely to be the most contaminated. This provides the greatest chance of detecting surface soil contamination. Adjacent areas will also be sampled to verify the initial expectation of which areas are contaminated and which are not.

3.3.1.1 UXO 5

Nineteen proposed composite surface soil samples (X5SS001 through X5SS019) will be collected at UXO 5 and submitted to a fixed-base laboratory for explosives and metals analyses. The soil samples will be used to help determine whether contamination is present within various areas at the site. Each composite soil sample will be comprised of four individual grab samples within an established grid pattern.

The lateral distance between individual aliquots in a composite sample will be on the order of a few feet. This is much closer than the distance between grab samples collected for the NSWC Crane basewide background study (TtNUS, 2001). Therefore, each composite sample will be viewed to be equivalent to the grab samples of the background study when comparing site and background metal concentrations.

UXO 5 has been separated into three areas and the sampling plans for each are presented below.

Drop Tower

Three composite surface soil samples will be collected around the 8- by 8-foot concrete drop pad where the 20-mm cartridges were drop tested. A grid of 8- by 9- foot rectangles will be marked off on three of

the four sides of the 8- by 8-foot concrete pad. The eastern side of the concrete pad abuts the concrete base of the drop tower itself, and therefore no samples will be collected in that area. Each rectangle will be bisected with string or flagging once vertically and once horizontally producing four distinct sample areas within each rectangle. One soil aliquot will be collected from the approximate center of each of those four sections and consolidated into one composite sample for fixed-base laboratory analysis. Additional composite samples will be collected in the outlying area of the concrete test pad. Figure 3-1 presents a drawing of all proposed surface soil sample locations and the areas they will represent. All soil borings will be advanced to a depth of 2 feet, and the soil samples will be analyzed as indicated in Table 3-1.

Rail Test Line

Eight composite surface soil samples will be collected along the test rail where CADs/PADs were used in the ejection seat testing process. A grid will be marked off on either side of the test rail running the length of the rail where the CADs/PADs were tested. Each grid will consist of a square shape approximately 10 feet by 10 feet. Within that square, the area will be bisected with string or flagging once vertically and once horizontally producing four distinct sample areas. One soil aliquot will be collected within each of those four sections and consolidated into one composite sample for fixed-base laboratory analysis. Additional composite samples will be collected in the outlying area of the test rail. Figure 3-1 presents all proposed surface soil sample locations and the areas they represent. All soil borings will be advanced to a depth of 2 feet, and the soil samples will be analyzed as indicated in Table 3-1.

Drainageway

Three composite soil samples will be collected within three individual 10 feet by 10 feet grid sections along the drainageway at the southern edge of the site. If there is a clear indication of soil deposition in the grassy drainageway, sample aliquots will be collected in the area of highest probable deposition. Figure 3-1 presents all proposed surface soil sample locations and the areas they represent. All soil borings will be advanced to a depth of 2 feet, and the soil samples will be analyzed as indicated in Table 3-1.

3.3.1.2 UXO 7

One hundred seventy-one grab surface soil samples are proposed to be collected at UXO 7 and analyzed for lead in the field using XRF equipment. All proposed sample locations are shown on Figure 3-2. Due to unknown exact locations of the ranges, actual sample locations may vary from the shown proposed locations. Final sample locations will be determined in the field during sample collection. However,

global positioning system points will be taken at each individual sample location which will allow for future repeatable investigations or guide in any remedial action. Upon collection of each surface soil sample, the soil will be visually inspected for the presence of lead shot. All visual observation results will be noted on the soil sample log. The sample will then be taken back to the field office and dried in a convection oven or similar device prior to XRF analyses. The results of the field XRF analyses will be the basis for determining which sample locations will be sent to the fixed-base laboratory for lead analysis and for correlation between laboratory and field results. At a minimum, 20 samples will be collected for XRF analysis within each individual site. Three separate XRF measurements will be made for each sample, and care will be taken to mix the sample in a plastic bag between measurements. The average of the three readings will be computed and recorded as the concentration value for the sample. Thirty-eight proposed surface soil samples at UXO 7 will be sent for laboratory analysis.

All soil borings will be logged to determine lithology which will determine the proper background data set to be used for comparison to the basewide background study.

The sampling plans for each of the sites that make up UXO 7 are presented below.

West Trap Range

Forty-four soil samples are proposed to be collected in the area of the West Trap Range and analyzed for lead in the field using XRF equipment. Prior to collection of the XRF samples, a site walkover of the West Trap Range will be conducted to assess whether any lead shot is visible on the surface or in the very near surface soils at various areas throughout the site. The FOL will use his professional judgment and inspection of the site to determine which areas are most likely to be contaminated with lead shot. The initial XRF sample(s) will then be collected in the area that comprised the shooters' assumed standing location. Based on a presumed shooting pattern, additional XRF samples will be collected in a fan-shaped pattern beginning approximately 40 feet north and northwest of the shooters' standing position and progressing into the apparently contaminated areas. This pattern and the distances presented here are based on firing range experience that indicates most lead shot is located at least 40 feet from the firing point. The actual samples for fixed-base laboratory analyses will then be determined based on these XRF sample concentrations. Samples will be selected for laboratory analysis to represent the entire range of XRF concentrations measured in the field at UXO 7. Figure 3-2 presents all proposed surface soil sample locations and the areas they represent at the West Trap Range. All soil borings will be advanced to a depth of 2 feet, and one soil sample from each boring will be analyzed as indicated in Table 3-1.

East Trap Range

Fifty-five soil samples are proposed to be collected in the area of the East Trap Range and analyzed for lead in the field using XRF equipment. This area includes sampling locations at the 100 yard and 200 yard shooting berms within the ORR shooting lanes. As with the West Trap Range, a site walkover of the East Trap Range will be conducted to assess whether any lead shot is visible on the surface or in the very near surface soils at various areas of the site prior to actual sample collection. The FOL will use his professional judgment and inspection of the site to determine which areas are most likely to be contaminated with lead shot. The initial XRF sample(s) will then be collected in the area that comprised the shooters' assumed standing location. Based on a presumed shooting pattern, additional XRF samples will be collected in a fan-shaped pattern beginning approximately 40 feet west of the shooters' standing position and progressing into the apparently contaminated areas. Samples will be selected for laboratory analysis to represent the entire range of XRF concentrations measured in the field at UXO 7. Figure 3-2 presents all proposed surface soil sample locations and the areas they represent at the East Trap Range. All soil borings will be advanced to a maximum depth of 2 feet, and one soil sample from each boring will be analyzed as indicated in Table 3-1.

South Pistol Range

Thirty-three soil samples are proposed to be collected in the area of the South Pistol Range and analyzed for lead in the field using XRF equipment. Samples will be collected in the three areas comprising the shooters' location as well as in and around the earthen berm located in the southwestern corner of the ORR, which was used as the backstop for the Pistol Range. Additional samples will be collected in the north facing hillside immediately south of the backstop where impact may have occurred due to missing the intended target area. Samples will be selected for laboratory analysis to represent the entire range of XRF concentrations measured in the field at UXO 7. Figure 3-2 presents all proposed surface soil sample locations and the areas they represent at the South Pistol Range. All soil borings will be advanced to a maximum depth of 2 feet, and one soil sample from each boring will be analyzed as indicated in Table 3-1.

ORR Shooting Lanes and Target Area

Thirty-nine soil samples are proposed to be collected along the ORR shooting lanes and target area and analyzed for lead in the field using XRF equipment. Several sample locations within the ORR shooting lanes are covered within the East Trap Range and therefore are not counted again here. Initial XRF samples will be collected at the 500 yard, 400 yard, and 300 yard earth berms used for firing, and the target area on the northern slopes and at the level ground at the base of the slopes. Additional samples

will be collected within the dirt mound which lies between the 400 and 300 yard shooting berms. This dirt mound is believed to post date all firing activities within the ORR. Samples will also be collected in the north facing hillside immediately south of the ORR target area where impact may have occurred due to missing the intended target area. Samples will be selected for laboratory analysis to represent the entire range of XRF concentrations measured in the field at UXO 7. Figure 3-2 presents all proposed surface soil sample locations and the areas they represent at the ORR shooting lanes and target area. All soil borings will be advanced to a maximum depth of 2 feet, and one soil sample from each boring will be analyzed as indicated in Table 3-1.

3.4 INVESTIGATION EQUIPMENT AND PROCEDURES

A hand auger will be used to collect surface soil samples to a depth of 2 feet. The procedure for soil sampling using hand auger techniques is included in SOP CTO034-08. Each removed soil core will be visually inspected and logged by the field geologist (see SOP CTO034-07), and the soil texture, grain size (sand, silt, or clay), color (and any unusual discoloration), moisture content, and soil type will be identified by the field geologist. Soil types will be based on the Unified Soil Classification System (USCS).

Duplicate soil samples will be collected at the rate presented in Section 3.5. Soil duplicates will be collected for those samples that have the greatest probability of containing contaminants (e.g., elevated levels of lead as indicated by XRF readings), but duplicates should not be collected for samples with concentrations exceeding 2,000 mg/kg because they are too different from typical risk-based concentration levels to be of practical value. The duplicate samples will be placed in the same types of containers and handled in the same manner as the regular soil samples. The duplicate samples will be given unique QC sample identifications (see SOP CTO034-02). Rinsate blanks will be collected as indicated on Table 3-2. For soil samples, the rinsate blank will consist of distilled water allowed to flow over dedicated decontaminated equipment. This process will continue until all the necessary bottles are filled for the rinsate sample.

3.4.1 Hand Auger Sampling for Surface Soil at UXO 5

The grab samples to be consolidated into composite samples will be mixed in a decontaminated stainless steel bowl using a decontaminated stainless steel spoon or dedicated disposable towel. Alternatively, the samples will be mixed in a 2-gallon disposable Ziploc bag. The appropriate sample jar(s) used for analyses will then be filled with the mixed composite samples and properly labeled. The hand auger bucket will be decontaminated in the field between each composite sample location. The bowl and spoon, if employed, will also be decontaminated in the field between each composite sample. Any used

Ziploc bags and disposable trowels will be collected in a trash bag and placed into an authorized dumpster for disposal. Details regarding the collection and labeling of soil samples are included in SOP CTO034-01. Soil sample analytical information is presented in Table 3-1, and appropriate containers to be used for each sample aliquot are listed in Table 3-3. After the samples are containerized, labeled, and bagged (see SOPs CTO034-01 and CTO034-03), they will be placed in a cooler containing ice until they can be packaged and prepared for shipment (Section 3.6.4). All soil borings will be backfilled with the remaining removed soil and if necessary, topped off with bentonite pellets to return the boring surface to surrounding ground surface. Any IDW will be handled in accordance with Section 3.10.

All individual sample aliquot locations will be marked with pin flags. The approximate center of each group of four aliquot locations will be identified with a tall wooden lath driven into the soil. The stake will have brightly colored flagging attached to it to increase visibility and will be labeled using a waterproof marker with a unique soil boring number corresponding to the boring log. This will be done so that the sampling location can be surveyed.

3.4.2 Hand Auger Sampling for Surface Soil at UXO 7

The locations of the 38 surface soil samples proposed for laboratory analysis at the individual sites that make up UXO 7 will be determined based on the field XRF analyses. The intent will be to represent all areas with lead concentrations greater than background concentrations. Soils collected for field XRF analysis will initially be placed in a Ziploc bag and transported to the field office where a visual inspection for lead shot will be done and results noted on the soil sample log. The sample aliquot will then be dried using a convection oven or similar device. After the XRF analysis, samples selected for fixed-base laboratory analysis will then be placed in the appropriate sample jar(s) and properly labeled. An attempt will be made to ensure that each sample submitted for laboratory analysis represents the same soil associated with the XRF results.

The hand auger bucket will be decontaminated in the field between each sample location. Details regarding the collection and labeling of soil samples are included in SOP CTO034-01. Soil sample analytical information is presented in Table 3-1, and appropriate containers to be used for each sample aliquot are listed in Table 3-3. After the samples are containerized, labeled, and bagged (see SOPs CTO034-01 and CTO034-03), they will be placed in a cooler containing ice until they can be packaged and prepared for shipment (Section 3.6.4).

All XRF sample locations will be backfilled with the remaining removed soil and if necessary, topped off with bentonite pellets to return the boring surface to surrounding ground surface. Any lead shot removed from the soil samples will be containerized and turned over to the NSWC Crane EDM. All other IDW will be handled in accordance with Section 3.10. When a boring has been sampled and backfilled, it will be identified by a tall wooden lath driven into the soil near the boring. The stake will have brightly colored flagging attached to it to increase visibility and will be labeled using a waterproof marker with a unique soil boring number corresponding to the boring log. This will be done so that the sampling location can be surveyed.

3.5 QUALITY CONTROL SAMPLES

This section focuses on field QC samples that will be collected as part of the RFAs, including field duplicates and equipment rinsate blanks. Table 3-2 presents the types and numbers of required field QC samples to be collected during the field investigations. Table 3-2 provides an overview of QC sample collection and analysis frequencies and corrective measures to be taken when the QC results are not within expectations. Field QC sample requirements for field duplicates and equipment rinsate blanks required for this project are described below.

Field Duplicates. Field duplicates are obtained during a single act of sampling and are used to assess the overall precision of the sampling and analysis program. Duplicate samples will be collected at a rate of one for every 20 samples of each type of environmental medium. All duplicate samples will be analyzed for the same parameters in the laboratory as their environmental sample counterparts.

Equipment Rinsate Blanks. Equipment rinsate blanks (or rinsate blanks) will be obtained under representative field conditions by running analyte-free water through sample collection equipment after decontamination and placing it in the appropriate sample containers for analysis. Equipment rinsate blanks will be collected for non-dedicated equipment. For surface soil sampling activities, rinsate blanks will be collected by running analyte-free water over the decontaminated non-dedicated sampling equipment before it is placed in sample jars.

3.6 SAMPLE HANDLING, PACKAGING, AND SHIPPING

3.6.1 Sample Preservation

Preservation requirements for each of the parameters of interest are provided in Table 3-3. All soil samples are to be cooled to 4 degrees Celsius ($^{\circ}\text{C}$) $\pm 2^{\circ}\text{C}$; no chemical preservatives are necessary. All samples will be promptly chilled with ice to $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ and packaged in an insulated cooler. Each cooler

will include a temperature blank. Ice will be sealed in containers to prevent water leakage. Samples will not be frozen. All XRF samples analyzed in the field will be maintained in the refrigerator located in the TtNUS field office until determined otherwise by TtNUS. If storage space does not allow this the project manager will be contacted to determine the most appropriate action. The intent is to retain samples in case additional samples are needed for correlation between laboratory and field concentrations.

3.6.2 Sample Labeling

Before samples are packaged, the sample labels will be checked to ensure that all information on the labels is complete and correct (see SOP CTO034-01) and that it matches the information on the sample collection log sheets and the Chain-of-Custody form.

3.6.3 Sample Packaging

Each sample container shipped to the laboratory will be placed in a Ziploc bag to prevent cross-contamination and leakage. If necessary, each Ziploc bag will be wrapped in bubblewrap to prevent breakage and cross-contamination. Only shipping containers that meet all applicable State and Federal standards for safe shipment will be used. Cube ice will be placed in and around the samples in sufficient quantity to ensure that the samples remain chilled ($4^{\circ}\text{C} \pm 2^{\circ}\text{C}$) during transport to the analytical laboratory.

SOP CTO034-01 provides a detailed description of sample handling, packaging, and shipping procedures required for this project. The FOL will be responsible for ensuring the completion of the following forms:

- Sample labels
- Chain-of-Custody forms
- Custody seals for coolers
- Shipping labels for coolers
- Express mail air bills

3.6.4 Sample Shipping

Shipping containers (i.e., coolers) will be sealed with nylon strapping tape in at least two places, and custody seals will be signed, dated, and affixed in a manner that will allow the receiver to quickly identify any tampering that may have occurred during transport to the laboratory (see SOPs CTO034-01 and -03).

Shipment will be made by a public courier at the next scheduled pickup following completion of sample collection. Copies of the air bills should be retained by the FOL for tracking purposes, if needed, and for communications with the laboratory. Air bills will be retained for the permanent record file.

3.6.5 Sample Custody

Custody of samples must be maintained and documented at all times, in accordance with SOP CTO034-03, beginning with the collection of samples in the field.

3.7 RECORD KEEPING

Standard forms, field notebooks, and a field logbook or electronic data files will be used to record all sample collection activities, field measurements, observations concerning site conditions, and other project-related information. Additional field records include sample log sheets, daily activity records, field logbooks, drilling and well completion log sheets, and field instrument calibration log sheets, among others. More details regarding record keeping are included in SOP CTO034-03.

All XRF data will initially be electronically logged in the XRF unit's Compaq IPAQ Pocket PC. The IPAQ will then be used to download the data into an Excel spreadsheet at the end of each day or more frequently, if possible.

3.7.1 Field Logbooks

Bound, weatherproof field notebooks or electronic daily activity logs will be maintained by sampling personnel. All information related to sampling and other field activities will be recorded in field notebooks or electronic daily activity logs. This information will include, but is not limited to, sampling time, weather conditions, unusual events, field measurements, and descriptions of photographs.

A bound, weatherproof logbook or electronic daily activity log will be maintained by the FOL. This book or log will contain a summary of each day's activities and will reference the field notebooks or electronic logs when applicable.

3.7.2 Equipment Calibration Logs

An equipment calibration log sheet will be used to record each time an instrument is calibrated, recalibrated, or checked against a standard or background. Field instruments will be calibrated in

accordance with the manufacturers procedures, and The XRF will be calibrated using National Institute of Standards and Technology traceable samples with low, moderate, and high concentrations of lead.

The procedures and standards to be used for instrument calibration are discussed in SOP CTO034-07.

3.7.5 Sample Collection Logs

All sample log sheets will be completed in accordance with SOP CTO0341-03.

3.7.6 Chain-of-Custody Forms

A Chain-of-Custody form will be completed for every cooler that contains samples being shipped to an off-site laboratory for analyses. These forms are a record of the people having custody of the samples from the time the samples are collected to the time they are analyzed and disposed (see SOP CTO034-03). The completed field Chain-of-custody document will be signed, placed in a sealed plastic envelope, and taped to the top inside lid of the shipping container before it is shipped. A copy of the Chain-of-Custody document will be retained by the FOL.

3.7.7 Shipping Forms/Air Bills

Copies of all forms and/or air bills related to the shipment of coolers will be retained by the FOL in order to trace the shipment, if necessary, and to communicate with the receiving laboratory.

3.7.8 Permanent Record File

At the completion of the field activities, the FOL will submit to the TOM all field records, data, field notebooks, logbooks, Chain-of-Custody records, sample log sheets, daily activity logs, and other records concerning the project, including all the forms and log sheets listed above. The FOL will check these records for legibility and completeness before submitting them to the TOM. These forms, data, and field notes will become part of the permanent project record.

3.8 SURVEYING

The location of every soil sample location will be marked with a wooden stake and flagging, and a boring hole number or sample ID will be marked on the stake.

The horizontal locations and vertical elevations of all soil sampling locations will be surveyed. For composite samples, the surveyed location will be the approximate center of the pattern formed by individual aliquot collection locations. It will be understood that this location represents an area larger than discrete locations typically associated with a single boring. Horizontal locations will be surveyed to the Indiana State Plane Coordinates System within the nearest foot and referenced to the 1983 North American Datum (NAD). The vertical elevations of the ground surface for the boring locations will be measured to the nearest 0.01 foot and referenced to the North American Vertical Datum (NAVD) of 1988.

3.9 EQUIPMENT DECONTAMINATION

All reusable equipment used to collect soil samples will be decontaminated in accordance with SOP CTO034-04.

3.10 MANAGEMENT OF INVESTIGATION-DERIVED WASTE

It is anticipated that this investigation will generate the following three types of potentially contaminated residues or investigation-derived waste (IDW):

- Personal protective equipment (PPE)
- Equipment decontamination fluids
- Waste cuttings from soil boring activities, including lead shot

IDW will be handled as described in SOP CTO034-09.

3.11 SAFETY

The health and safety activities described in the following sections will ensure that field activities are performed at a high level of safety.

3.11.1 Health and Safety Plan

The existing HASP used for the RFI at SWMU 7 has been updated and modified to include the additional work at UXO 5 and UXO 7. The HASP has been prepared to describe specific health and safety requirements, concerns, and information related to these site activities. This document will be read and understood by each person working at the site. Each worker or visitor to the site must sign an acknowledgment that he or she has read and understands the HASP.

3.11.2 Health and Safety Training

All workers involved with the site assessments shall have successfully completed the OSHA-mandated 40-hour health and safety training and the follow-up annual 8-hour refresher courses when appropriate.

TtNUS and subcontractor personnel will supply OSHA documentation to the FOL before beginning work. Personnel who do not comply with this requirement must receive verbal approval to work from TtNUS corporate health and safety personnel.

3.11.3 Medical Approval and Personal Protective Clothing and Equipment

Workers at the site must be part of a medical monitoring program and must be medically approved to perform their duties without physical limitations. Protective clothing and equipment, as specified in the HASP, will be worn while performing site activities.

3.11.4 Safety Meetings

Safety meetings will be held among on-site workers whenever it is deemed appropriate by the SSO. The SSO will discuss safety issues related to activities being performed and will make site workers aware of any new conditions that could potentially affect health or safety.

3.12 ORGANIZATION AND LOGISTICS OF FIELD INVESTIGATIONS

3.12.1 Personnel

The duties, responsibilities, and lines of command for each person working on the project are described in Section 2.0 and displayed on Figures 2-1 and 2-2. Persons working on the project will be intimately familiar with their roles and responsibilities. In addition, they will be familiar with the mechanisms and procedures for coordinating tasks, improving communications, and reporting incidences or irregularities. The FOL is responsible for coordinating all on-site personnel and activities (Section 2.3.1). The SSO is responsible for health and safety monitoring and ensures that the HASP is adhered to during all field activities (Section 2.3.3). The SSO has the authority to stop work if an imminent safety hazard is encountered (Section 2.3.3).

3.12.2 Subcontractors

The FOL will direct all surveying subcontractor activities on site and the Project Chemist will direct the activities of the fixed-base laboratory.

3.12.3 Mobilization and Demobilization

Following approval of the QAPP, TtNUS will begin mobilization activities. All field team members will review the QAPP and HASP prior to mobilizing to the field. In addition, a field team orientation meeting will be held to familiarize personnel with the scope of the field activities. Items to be presented during that meeting include the following:

- Identification of the QAPP Addendum No. 2, the HASP, and applicable field SOPs (Appendix A).
- Site-specific safety concerns and requirements.
- Project objectives.
- Sampling designs and strategies for soil (including the relationship of soils data for this project to NSWC Crane background soils data).
- Site-specific particulars of field operations (e.g., locations of utilities, physical access to sampling locations, communication mechanisms, lines of authority and responsibility, scheduling requirements, sample shipping concerns, etc.).
- Laboratory and other subcontractor coordination.
- Site access requirements.
- Travel requirements.

The FOL will coordinate the mobilization activities for this project. The equipment required for the field activities will be mobilized from the TtNUS Pittsburgh office or third-party vendor(s). The FOL and crew will demobilize from the site upon completion of the field operations and transport field equipment back to the TtNUS Pittsburgh office, as necessary. All work areas will be thoroughly checked, and trash will be removed and disposed properly.

TABLE 3-1

LISTING OF SOIL SAMPLES AND LABORATORY ANALYSES
UXO 5 AND UXO 7 QAPP ADDENDUM NO. 2
NSWC CRANE
CRANE, INDIANA
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Note: The rationale for collecting and analyzing samples as indicated in this Table is to determine the presence or absence of explosives (UXO 5), TAL Metals (UXO 5), and Lead (UXO 7) and to support a preliminary risk evaluation.

Sample Location ¹	Sample ID ²	Explosives	TAL Metals		
		Nitroaromatics and Nitramines SW-846 8330	SW-846 3050B/6020 (TAL Metals)	SW-846 3050B/6020 (Lead Only)	Field XRF (Lead Only)
UXO 5					
X5SB001	X5SS001C0002	1	1	NA	NA
X5SB002	X5SS002C0002	1	1	NA	NA
X5SB003	X5SS003C0002	1	1	NA	NA
X5SB004	X5SS004C0002	1	1	NA	NA
X5SB005	X5SS005C0002	1	1	NA	NA
X5SB006	X5SS006C0002	1	1	NA	NA
X5SB007	X5SS007C0002	1	1	NA	NA
X5SB008	X5SS008C0002	1	1	NA	NA
X5SB009	X5SS009C0002	1	1	NA	NA
X5SB010	X5SS010C0002	1	1	NA	NA
X5SB011	X5SS011C0002	1	1	NA	NA
X5SB012	X5SS012C0002	1	1	NA	NA
X5SB013	X5SS013C0002	1	1	NA	NA
X5SB014	X5SS014C0002	1	1	NA	NA
X5SB015	X5SS015C0002	1	1	NA	NA
X5SB016	X5SS016C0002	1	1	NA	NA
X5SB017	X5SS017C0002	1	1	NA	NA
X5SB018	X5SS018C0002	1	1	NA	NA
X5SB019	X5SS019C0002	1	1	NA	NA
Total Soil Samples - UXO 5		19	19		0
UXO 7					
X7SB001	X7SS001G0002	NA	NA	TBD ³	1
X7SB002	X7SS002G0002	NA	NA	TBD ³	1
X7SB003	X7SS003G0002	NA	NA	TBD ³	1
X7SB004	X7SS004G0002	NA	NA	TBD ³	1
X7SB005	X7SS005G0002	NA	NA	TBD ³	1
X7SB006	X7SS006G0002	NA	NA	TBD ³	1
X7SB007	X7SS007G0002	NA	NA	TBD ³	1
X7SB008	X7SS008G0002	NA	NA	TBD ³	1
X7SB009	X7SS009G0002	NA	NA	TBD ³	1
X7SB010	X7SS010G0002	NA	NA	TBD ³	1
X7SB011	X7SS011G0002	NA	NA	TBD ³	1
X7SB012	X7SS012G0002	NA	NA	TBD ³	1

TABLE 3-1

**LISTING OF SOIL SAMPLES AND LABORATORY ANALYSES
UXO 5 AND UXO 7 QAPP ADDENDUM NO. 2**

**NSWC CRANE
CRANE, INDIANA**

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Sample Location ¹	Sample ID ²	Explosives	TAL Metals		
		Nitroaromatics and Nitramines SW-846 8330	SW-846 3050B/6020 (TAL Metals)	SW-846 3050B/6020 (Lead Only)	Field XRF (Lead Only)
X7SB013	X7SS013G0002	NA	NA	TBD ³	1
X7SB014	X7SS014G0002	NA	NA	TBD ³	1
X7SB015	X7SS015G0002	NA	NA	TBD ³	1
X7SB016	X7SS016G0002	NA	NA	TBD ³	1
X7SB017	X7SS017G0002	NA	NA	TBD ³	1
X7SB018	X7SS018G0002	NA	NA	TBD ³	1
X7SB019	X7SS019G0002	NA	NA	TBD ³	1
X7SB020	X7SS020G0002	NA	NA	TBD ³	1
X7SB021	X7SS021G0002	NA	NA	TBD ³	1
X7SB022	X7SS022G0002	NA	NA	TBD ³	1
X7SB023	X7SS023G0002	NA	NA	TBD ³	1
X7SB024	X7SS024G0002	NA	NA	TBD ³	1
X7SB025	X7SS025G0002	NA	NA	TBD ³	1
X7SB026	X7SS026G0002	NA	NA	TBD ³	1
X7SB027	X7SS027G0002	NA	NA	TBD ³	1
X7SB028	X7SS028G0002	NA	NA	TBD ³	1
X7SB029	X7SS029G0002	NA	NA	TBD ³	1
X7SB030	X7SS030G0002	NA	NA	TBD ³	1
X7SB031	X7SS031G0002	NA	NA	TBD ³	1
X7SB032	X7SS032G0002	NA	NA	TBD ³	1
X7SB033	X7SS033G0002	NA	NA	TBD ³	1
X7SB034	X7SS034G0002	NA	NA	TBD ³	1
X7SB035	X7SS035G0002	NA	NA	TBD ³	1
X7SB036	X7SS036G0002	NA	NA	TBD ³	1
X7SB037	X7SS037G0002	NA	NA	TBD ³	1
X7SB038	X7SS038G0002	NA	NA	TBD ³	1
X7SB039	X7SS039G0002	NA	NA	TBD ³	1
X7SB040	X7SS040G0002	NA	NA	TBD ³	1
X7SB041	X7SS041G0002	NA	NA	TBD ³	1
X7SB042	X7SS042G0002	NA	NA	TBD ³	1
X7SB043	X7SS043G0002	NA	NA	TBD ³	1
X7SB044	X7SS044G0002	NA	NA	TBD ³	1
X7SB045	X7SS045G0002	NA	NA	TBD ³	1
X7SB046	X7SS046G0002	NA	NA	TBD ³	1
X7SB047	X7SS047G0002	NA	NA	TBD ³	1

TABLE 3-1

LISTING OF SOIL SAMPLES AND LABORATORY ANALYSES
UXO 5 AND UXO 7 QAPP ADDENDUM NO. 2
NSWC CRANE
CRANE, INDIANA
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Sample Location ¹	Sample ID ²	Explosives	TAL Metals		
		Nitroaromatics and Nitramines SW-846 8330	SW-846 3050B/6020 (TAL Metals)	SW-846 3050B/6020 (Lead Only)	Field XRF (Lead Only)
X7SB048	X7SS048G0002	NA	NA	TBD ³	1
X7SB049	X7SS049G0002	NA	NA	TBD ³	1
X7SB050	X7SS050G0002	NA	NA	TBD ³	1
X7SB051	X7SS051G0002	NA	NA	TBD ³	1
X7SB052	X7SS052G0002	NA	NA	TBD ³	1
X7SB053	X7SS053G0002	NA	NA	TBD ³	1
X7SB054	X7SS054G0002	NA	NA	TBD ³	1
X7SB055	X7SS055G0002	NA	NA	TBD ³	1
X7SB056	X7SS056G0002	NA	NA	TBD ³	1
X7SB057	X7SS057G0002	NA	NA	TBD ³	1
X7SB058	X7SS058G0002	NA	NA	TBD ³	1
X7SB059	X7SS059G0002	NA	NA	TBD ³	1
X7SB060	X7SS060G0002	NA	NA	TBD ³	1
X7SB061	X7SS061G0002	NA	NA	TBD ³	1
X7SB062	X7SS062G0002	NA	NA	TBD ³	1
X7SB063	X7SS063G0002	NA	NA	TBD ³	1
X7SB064	X7SS064G0002	NA	NA	TBD ³	1
X7SB065	X7SS065G0002	NA	NA	TBD ³	1
X7SB066	X7SS066G0002	NA	NA	TBD ³	1
X7SB067	X7SS067G0002	NA	NA	TBD ³	1
X7SB068	X7SS068G0002	NA	NA	TBD ³	1
X7SB069	X7SS069G0002	NA	NA	TBD ³	1
X7SB070	X7SS070G0002	NA	NA	TBD ³	1
X7SB071	X7SS071G0002	NA	NA	TBD ³	1
X7SB072	X7SS072G0002	NA	NA	TBD ³	1
X7SB073	X7SS073G0002	NA	NA	TBD ³	1
X7SB074	X7SS074G0002	NA	NA	TBD ³	1
X7SB075	X7SS075G0002	NA	NA	TBD ³	1
X7SB076	X7SS076G0002	NA	NA	TBD ³	1
X7SB077	X7SS077G0002	NA	NA	TBD ³	1
X7SB078	X7SS078G0002	NA	NA	TBD ³	1
X7SB079	X7SS079G0002	NA	NA	TBD ³	1
X7SB080	X7SS080G0002	NA	NA	TBD ³	1
X7SB081	X7SS081G0002	NA	NA	TBD ³	1
X7SB082	X7SS082G0002	NA	NA	TBD ³	1

TABLE 3-1

**LISTING OF SOIL SAMPLES AND LABORATORY ANALYSES
UXO 5 AND UXO 7 QAPP ADDENDUM NO. 2**

**NSWC CRANE
CRANE, INDIANA**

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Sample Location ¹	Sample ID ²	Explosives	TAL Metals		
		Nitroaromatics and Nitramines SW-846 8330	SW-846 3050B/6020 (TAL Metals)	SW-846 3050B/6020 (Lead Only)	Field XRF (Lead Only)
X7SB083	X7SS083G0002	NA	NA	TBD ³	1
X7SB084	X7SS084G0002	NA	NA	TBD ³	1
X7SB085	X7SS085G0002	NA	NA	TBD ³	1
X7SB086	X7SS086G0002	NA	NA	TBD ³	1
X7SB087	X7SS087G0002	NA	NA	TBD ³	1
X7SB088	X7SS088G0002	NA	NA	TBD ³	1
X7SB089	X7SS089G0002	NA	NA	TBD ³	1
X7SB090	X7SS090G0002	NA	NA	TBD ³	1
X7SB091	X7SS091G0002	NA	NA	TBD ³	1
X7SB092	X7SS092G0002	NA	NA	TBD ³	1
X7SB093	X7SS093G0002	NA	NA	TBD ³	1
X7SB094	X7SS094G0002	NA	NA	TBD ³	1
X7SB095	X7SS095G0002	NA	NA	TBD ³	1
X7SB096	X7SS096G0002	NA	NA	TBD ³	1
X7SB097	X7SS097G0002	NA	NA	TBD ³	1
X7SB098	X7SS098G0002	NA	NA	TBD ³	1
X7SB099	X7SS099G0002	NA	NA	TBD ³	1
X7SB100	X7SS100G0002	NA	NA	TBD ³	1
X7SB101	X7SS101G0002	NA	NA	TBD ³	1
X7SB102	X7SS102G0002	NA	NA	TBD ³	1
X7SB103	X7SS103G0002	NA	NA	TBD ³	1
X7SB104	X7SS104G0002	NA	NA	TBD ³	1
X7SB105	X7SS105G0002	NA	NA	TBD ³	1
X7SB106	X7SS106G0002	NA	NA	TBD ³	1
X7SB107	X7SS107G0002	NA	NA	TBD ³	1
X7SB108	X7SS108G0002	NA	NA	TBD ³	1
X7SB109	X7SS109G0002	NA	NA	TBD ³	1
X7SB110	X7SS110G0002	NA	NA	TBD ³	1
X7SB111	X7SS111G0002	NA	NA	TBD ³	1
X7SB112	X7SS112G0002	NA	NA	TBD ³	1
X7SB113	X7SS113G0002	NA	NA	TBD ³	1
X7SB114	X7SS114G0002	NA	NA	TBD ³	1
X7SB115	X7SS115G0002	NA	NA	TBD ³	1
X7SB116	X7SS116G0002	NA	NA	TBD ³	1
X7SB117	X7SS117G0002	NA	NA	TBD ³	1

TABLE 3-1

**LISTING OF SOIL SAMPLES AND LABORATORY ANALYSES
UXO 5 AND UXO 7 QAPP ADDENDUM NO. 2**

**NSWC CRANE
CRANE, INDIANA**

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Sample Location ¹	Sample ID ²	Explosives	TAL Metals		
		Nitroaromatics and Nitramines SW-846 8330	SW-846 3050B/6020 (TAL Metals)	SW-846 3050B/6020 (Lead Only)	Field XRF (Lead Only)
X7SB118	X7SS118G0002	NA	NA	TBD ³	1
X7SB119	X7SS119G0002	NA	NA	TBD ³	1
X7SB120	X7SS120G0002	NA	NA	TBD ³	1
X7SB121	X7SS121G0002	NA	NA	TBD ³	1
X7SB122	X7SS122G0002	NA	NA	TBD ³	1
X7SB123	X7SS123G0002	NA	NA	TBD ³	1
X7SB124	X7SS124G0002	NA	NA	TBD ³	1
X7SB125	X7SS125G0002	NA	NA	TBD ³	1
X7SB126	X7SS126G0002	NA	NA	TBD ³	1
X7SB127	X7SS127G0002	NA	NA	TBD ³	1
X7SB128	X7SS128G0002	NA	NA	TBD ³	1
X7SB129	X7SS129G0002	NA	NA	TBD ³	1
X7SB130	X7SS130G0002	NA	NA	TBD ³	1
X7SB131	X7SS131G0002	NA	NA	TBD ³	1
X7SB132	X7SS132G0002	NA	NA	TBD ³	1
X7SB133	X7SS133G0002	NA	NA	TBD ³	1
X7SB134	X7SS134G0002	NA	NA	TBD ³	1
X7SB135	X7SS135G0002	NA	NA	TBD ³	1
X7SB136	X7SS136G0002	NA	NA	TBD ³	1
X7SB137	X7SS137G0002	NA	NA	TBD ³	1
X7SB138	X7SS138G0002	NA	NA	TBD ³	1
X7SB139	X7SS139G0002	NA	NA	TBD ³	1
X7SB140	X7SS140G0002	NA	NA	TBD ³	1
X7SB141	X7SS141G0002	NA	NA	TBD ³	1
X7SB142	X7SS142G0002	NA	NA	TBD ³	1
X7SB143	X7SS143G0002	NA	NA	TBD ³	1
X7SB144	X7SS144G0002	NA	NA	TBD ³	1
X7SB145	X7SS145G0002	NA	NA	TBD ³	1
X7SB146	X7SS146G0002	NA	NA	TBD ³	1
X7SB147	X7SS147G0002	NA	NA	TBD ³	1
X7SB148	X7SS148G0002	NA	NA	TBD ³	1
X7SB149	X7SS149G0002	NA	NA	TBD ³	1
X7SB150	X7SS150G0002	NA	NA	TBD ³	1
X7SB151	X7SS151G0002	NA	NA	TBD ³	1
X7SB152	X7SS152G0002	NA	NA	TBD ³	1

TABLE 3-1

**LISTING OF SOIL SAMPLES AND LABORATORY ANALYSES
UXO 5 AND UXO 7 QAPP ADDENDUM NO. 2**

**NSWC CRANE
CRANE, INDIANA**

PAGE 6 OF 6

Sample Location ¹	Sample ID ²	Explosives	TAL Metals		
		Nitroaromatics and Nitramines SW-846 8330	SW-846 3050B/6020 (TAL Metals)	SW-846 3050B/6020 (Lead Only)	Field XRF (Lead Only)
X7SB153	X7SS153G0002	NA	NA	TBD ³	1
X7SB154	X7SS154G0002	NA	NA	TBD ³	1
X7SB155	X7SS155G0002	NA	NA	TBD ³	1
X7SB156	X7SS156G0002	NA	NA	TBD ³	1
X7SB157	X7SS157G0002	NA	NA	TBD ³	1
X7SB158	X7SS158G0002	NA	NA	TBD ³	1
X7SB159	X7SS159G0002	NA	NA	TBD ³	1
X7SB160	X7SS160G0002	NA	NA	TBD ³	1
X7SB161	X7SS161G0002	NA	NA	TBD ³	1
X7SB162	X7SS162G0002	NA	NA	TBD ³	1
X7SB163	X7SS163G0002	NA	NA	TBD ³	1
X7SB164	X7SS164G0002	NA	NA	TBD ³	1
X7SB165	X7SS165G0002	NA	NA	TBD ³	1
X7SB166	X7SS166G0002	NA	NA	TBD ³	1
X7SB167	X7SS167G0002	NA	NA	TBD ³	1
X7SB168	X7SS168G0002	NA	NA	TBD ³	1
X7SB169	X7SS169G0002	NA	NA	TBD ³	1
X7SB170	X7SS170G0002	NA	NA	TBD ³	1
X7SB171	X7SS171G0002	NA	NA	TBD ³	1
Total Soil Samples - UXO 7		0	0	0	171

C = Composite sample

G = Grab sample

NA = Not applicable

1 X = UXO

2 Sample depth to a maximum of 24 inches.

3 Depending on field XRF screenings, up to 38 samples will be selected for fixed-based laboratory analysis.

TABLE 3-2

**SUMMARY OF SOIL ANALYSES AND QUALITY CONTROL SAMPLES
UXO 5 AND UXO 7 QAPP ADDENDUM NO. 2
NSWC CRANE
CRANE, INDIANA**

Sample Type	Explosives	Metals		
	Nitroaromatics and Nitramines SW-846 8330	SW-846 3050B/6020 (TAL Metals)	SW-846 3050B/6020 (Lead only)	Field XRF (Lead only)
Total Samples (UXO 5 and 7)	19	19	38	171
Field Duplicates ⁽¹⁾	1	1	2	9
Rinsate Blanks ⁽²⁾	1	1	1	1
Matrix Spikes ⁽³⁾	1	1	2	9
Matrix Spike Duplicates ⁽⁴⁾	1	1	2	9

1) Field duplicates will be collected at a frequency of one per every 20 environmental samples.

2) Rinsate blanks will be collected at a frequency of one per sampling device or instrument. These amounts are estimates and may vary.

3) Matrix spikes are collected for all organic and inorganic parameters at a frequency of one per every 20 field

4) Matrix spike duplicates are collected for all organic parameters. Laboratory duplicates are analyzed for inorganic parameters in lieu of matrix spike duplicates. Matrix spike duplicates are collected at a frequency of one per every 20 field samples.

NA - Not applicable

QAPP - Quality Assurance Project Plan

TABLE 3-3

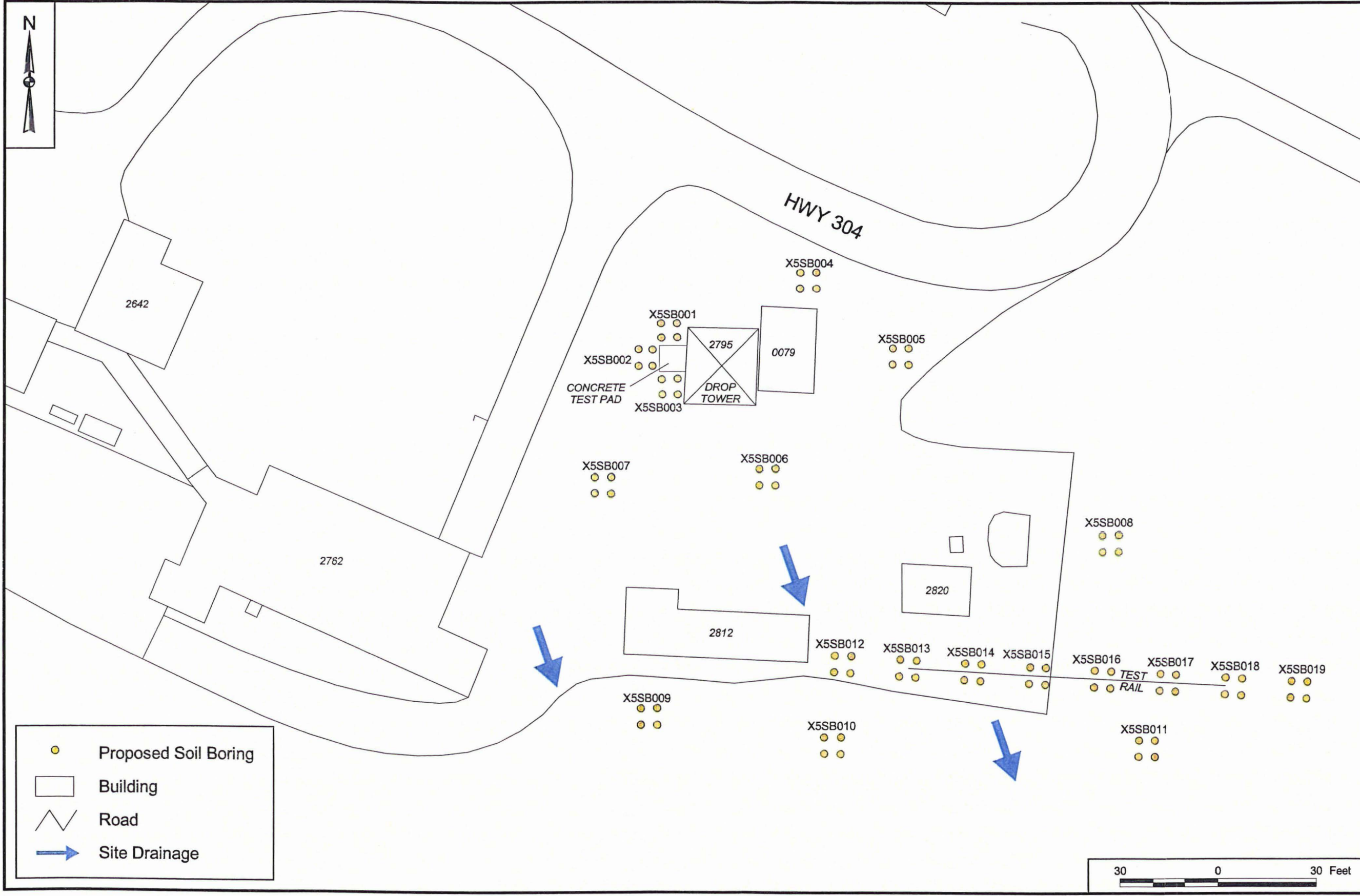
**SUMMARY OF SAMPLE ANALYSES, CONTAINER TYPES AND VOLUMES,
PRESERVATION REQUIREMENTS, AND HOLDING TIMES FOR SOILS
UXO 5 AND UXO 7 QAPP ADDENDUM NO. 2
NSWC CRANE
CRANE, INDIANA**

Parameter	Analyte/Methodology	Sample Container	Container Volume ⁽¹⁾	Preservation	Maximum Holding Time ⁽²⁾
Explosives (Nitroaromatics and Nitramines)	SW-846 8330	Wide-mouth jar, Teflon- lined plastic cap	(1) 4 ounce	Cool to 4 °C, dark	Extraction within 7 days; analysis within 40 days of extraction.
Metals (including lead only)	SW-846 3050B/6020 (7471A)	Wide-mouth jar, Teflon- lined plastic cap	(1) 4 ounce	Cool to 4 °C, dark	180 days to analysis except mercury (28 days to analysis)

1 The number in parenthesis indicates the sample container quantity.

2 All holding times are from date of collection.

°C - Degrees Celsius



CONTRACT NO. CTO 0034		APPROVED BY		DATE	
APPROVED BY		APPROVED BY		DATE	
DRAWING NO.		FIGURE 3 - 1		REV	
UXO 5		PROPOSED SURFACE SOIL SAMPLING LOCATIONS		0	
UXO 5/UXO 7 QAPP ADDENDUM NO. 2		NSWC CRANE			
		CRANE, INDIANA			
DATE 1/17/07		DATE 1/18/07		SCALE	
DRAWN BY K MOORE		CHECKED BY J. GOERT		AS NOTED	
		COST/SCHED-AREA			



DRAWN BY	DATE
K. MOORE	3/5/07
CHECKED BY	DATE
J. GOERDT	3/7/07
COST/SCHEDULE-AREA	
SCALE AS NOTED	



UXO 7 XRF SAMPLE LOCATIONS
2003 AERIAL PHOTOGRAPHY
UXO 5/UXO 7 QAPP ADDENDUM NO. 2
NSWC CRANE
CRANE, INDIANA

CONTRACT NUMBER 0034	
APPROVED BY	DATE
APPROVED BY	DATE
DRAWING NO. FIGURE 3 - 2	REV 0

4.0 LABORATORY OPERATIONS AND DATA REDUCTION, REVIEW, AND MANAGEMENT

4.1 SAMPLE CUSTODY PROCEDURES

Samples will be labeled and shipped to the analytical laboratory in accordance with Section 3.0 of this document. Laboratory-specific custody procedures are governed by Section 4.0 of the SWMUs 8 and 15 QAPP (TtNUS, 2004).

4.2 LABORATORY INSTRUMENT CALIBRATION PROCEDURES AND FREQUENCIES

Refer to Section 5.2 of the original SWMUs 8 and 15 QAPP (TtNUS, 2004) for laboratory calibration requirements.

4.3 LABORATORY ANALYTICAL AND MEASUREMENT PROCEDURES

4.3.1 Sample Analyses

The laboratory analyses of confirmation samples will be performed by Laucks Testing Laboratories. Laucks Testing Laboratories has successfully completed the laboratory evaluation process required as part of the Naval Facilities Engineering Services Command (NFESC) QA Program and described in the Navy Installation Restoration Laboratory Quality Assurance Guide (NFESC, 1996). Table 3-1 provides a summary of the laboratory analytical methods. Laboratory SOPs are identified in Appendix B.

4.3.2 List of Project Target Compounds and Detection Limits

A list of the laboratory target analytes and laboratory-specific target values (TVs) for organic compounds, instrument detection limits (IDLs) for metals, and reporting limits (RLs) for all applicable analytes is provided in Table 1-5 of the SWMUs 8 and 15 QAPP (TtNUS, 2004). Table 3-1 of this QAPP Addendum No. 2 identifies the analyses to be conducted. The analytical fraction (i.e., lead) corresponds to the same entries in Table 1-5 of TtNUS (2004).

4.4 LABORATORY QUALITY CONTROL CHECKS

Descriptions of various data quality indicators (DQIs) and the types of laboratory QC samples used to measure or verify DQIs are presented in Sections 7.0 through 7.5 of SWMUs 8 and 15 QAPP (TtNUS, 2004). Requirements for laboratory QC checks are presented in Section 7.8 of TtNUS (2004). A list of

the applicable non-calibration QC requirements for confirmation sample analyses is presented in Table 4-1 of this QAPP Addendum No. 2.

4.5 DATA REDUCTION, VALIDATION, ASSESSMENT, AND REPORTING

Refer to Section 8.0 of the SWMU 8 and 15 QAPP (TtNUS, 2004) for laboratory-specific requirements. Section 8.0 of TtNUS (2004) also identifies requirements for reconciling data against DQOs.

4.5.1 Field Data Reduction and Comparisons

Upon validation, laboratory lead concentrations will be compared to field XRF lead concentrations through a correlation plot. The field data will be plotted against the laboratory data, and a linear least-squares fit will be applied to generate an estimate of the "best" line through the data. If the data set exhibits more than one region where there appears to be a linear fit to the data but the regions have different slopes (this has occurred at NSWC Crane in the past), the data may be subdivided into separate regions for fitting. No more than three separate regions of the correlation will be fit in this manner, and as few regions as possible will be used to adequately represent a linear fit. If a correlation coefficient (commonly denoted as " r ") of 0.65 or greater is obtained for a region, the fit will be considered adequate enough to translate field XRF results to the equivalent laboratory lead concentrations with confidence. A correlation coefficient less than 0.65 will be considered unacceptable for making this translation. An ideal correlation coefficient would be 1.0.

Assuming a satisfactory correlation coefficient is obtained, the field XRF data will be translated to the equivalent laboratory concentrations, which will then be used for estimating the spatial distribution and magnitude of lead contamination in surface soil. If the correlation coefficient is less than 0.65, several courses of action are possible. One of which may include fixed-base laboratory analysis of additional temperature controlled samples that will be stored on-site. A second option may include adjustments or modifications during design of a remediation plan, if remediation is necessary. In any case, the Navy will consult with the U.S. EPA as to the appropriate course of action.

4.5.2 Laboratory Data Reduction and Reporting

Laboratory data reduction will be conducted in accordance with the laboratory SOPs provided in Appendix B. The reported data shall follow the requirements of the laboratory specification, which shall require that a Contract Laboratory Program (CLP)-like data package be provided to TtNUS upon

completion of the analyses. Electronic data shall be reported in accordance with current TtNUS electronic data deliverable requirements as presented in the laboratory specification.

4.6 LABORATORY PERFORMANCE AND SYSTEM AUDITS

Laboratory performance and system audit requirements are presented in Section 9.2 of the SMWUs 8 and 15 QAPP (TtNUS, 2004).

4.7 LABORATORY INSTRUMENT PREVENTIVE MAINTENANCE

Proper maintenance of laboratory instruments and equipment is essential. Depending on manufacturers' recommendations, maintenance intervals are established for each instrument. All instruments will be labeled with a model number and serial number, and a maintenance logbook will be maintained for each instrument. Personnel will be alert to the maintenance status of the equipment they are using at all times.

4.8 LABORATORY CORRECTIVE ACTION

In general, laboratory corrective actions are warranted whenever an out-of-control event or potential out-of-control event is noted. The specific corrective action taken depends on the specific analysis and the nature of the event. Generally, the following occurrences alert laboratory personnel that corrective action may be necessary:

- QC data are outside established warning or control limits.
- Method blank analyses yield concentrations of target analytes greater than acceptable levels.
- Undesirable trends are detected in spike percent recoveries (%Rs) or in duplicate relative percent difference (RPDs).
- There is an unexplained change in compound detection capability.
- Inquiries concerning data quality are received.
- Deficiencies are detected by laboratory QA staff during audits or from performance evaluation (PE) sample test results.

4.8.1 Corrective Action Documentation

Procedures for laboratory corrective actions are specified in the associated laboratory SOPs. Any corrective action taken above the analyst level that cannot be performed immediately at the instrument will be documented.

Laboratory corrective actions must be documented and included as part of the final evidence file. The TtNUS TOM will be informed of all major corrective actions that do not bring DQO-related nonconformances into conformance with project DQOs. The TtNUS TOM will advise all levels of project management in accordance with Section 12.0 of the SWMU 8s and 15 QAPP (TtNUS, 2004).

4.8.2 Corrective Action During Data Validation and Data Assessment

The need for corrective action may become apparent during data validation, interpretation, or presentation activities. Rework (i.e., resampling or reanalysis), a change in work procedures, or additional or refresher training are possible corrective actions relevant to data evaluation activities. The TOM will be responsible for approving the implementation of a corrective action and ensuring that it is documented appropriately for all deficiencies. Analytical data may be qualified during data validation to alert data users of the potential that particular analytical results are potentially deficient relative to expected performance standards. Such validation practices are described in Section 8.0 of the SWMUs 8 and 15 QAPP (TtNUS, 2004). When conducting data assessment for project decision making, a number of situation-dependent qualifications of data or decisions are possible. The number of possible situations or conditions precludes enumeration of all possible corrective actions; however, the approach used to identify and impose such qualifications is described in Section 8.0 of TtNUS (2004).

4.9 QUALITY ASSURANCE REPORTS TO MANAGEMENT

QA reports to management will be provided in four primary formats during the course of this investigation: data validation reports, reports summarizing accomplishments and QA/QC issues during the field investigation, project-wide progress reports, and laboratory QA reports. The report frequencies, content, preparers, and recipients are summarized in Table 12-1 of the SWMUs 8 and 15 QAPP (TtNUS, 2004).

Data validation reports for confirmation samples will address all major and minor laboratory noncompliances, as well as noted sample matrix effects. In the event that major problems occur with the analytical laboratory (e.g., repeated or extreme holding time exceedances or calibration noncompliances, etc.), the data validation manager will notify the TOM, QAM, PM, technical coordinator, and laboratory services coordinator. Such notifications (if necessary) are typically provided via internal memoranda and

are placed in the project file. These reports contain a summary of the noncompliance, a synopsis of the impact on individual projects, and recommendations regarding corrective action and compensation adjustments. Corrective actions for major noncompliances are initiated at the program level.

The TtNUS FOL will provide the TtNUS PM and TOM with daily verbal field progress reports during the course of the sampling events. These reports will explain accomplishments, deviations from the QAPP, and upcoming activities, and will provide a QA summary. The TOM will provide a monthly progress report to the Navy that addresses the project budget, schedule, accomplishments, planned activities, and QA/QC issues and intended corrective actions.

The analytical laboratory for confirmation analyses will provide QA reports to TtNUS if QC limits are updated or if other significant plan deviations result from unanticipated circumstances. Because method detection limits (MDLs) will be included in the analytical data packages for confirmation, it is not necessary for the laboratories to include updated MDLs in their QA reports unless the updates result in MDLs that exceed cleanup levels applicable to this project.

4.10 FINAL EVIDENCE FILES

Refer to Section 4.3 of the SWMUs 8 and 15 QAPP (TtNUS, 2004) for requirements governing the management of project records up to and including final disposition.

TABLE 4-1

**NON-CALIBRATION QC SAMPLE USAGE FREQUENCIES,
ACCEPTANCE LIMITS, AND CORRECTIVE ACTIONS
UXO 5 AND UXO 7 QAPP ADDENDUM NO. 2**

**NSWC CRANE
CRANE, INDIANA
PAGE 1 OF 2**

QC Sample Type	Collection Frequency	Acceptance Limits	Corrective Action
Field Duplicate	One per 20 investigative samples collected.	Aqueous = NA Soil/Sediment = 70% RPD	Qualify data according to data validation requirements.
Equipment Rinsate Blank	One per non-dedicated sampling device.	Less than RL	Laboratory action taken per LTL-1008. TtNUS action taken per validation protocols and Section 11.3 of SWMUs 8 and 15 QAPP (TtNUS, 2004).
Source Water Blank	One per water source used for decontamination.	NA	NA
Internal Standard	At least one internal standard per sample for GC/MS analysis.	Retention times stable to ± 30 seconds; area counts stable to within factor of two	Laboratory action taken per LTL-1008. TtNUS action taken per validation protocols and Section 11.3 of SWMUs 8 and 15 QAPP (TtNUS, 2004).
Laboratory Control Sample	One per 20 environmental samples per matrix per analytical fraction.	See Tables 7-1 and 7-2 of TtNUS (2004)	Laboratory action taken per LTL-1008. TtNUS action taken per validation protocols and Section 11.3 of SWMUs 8 and 15 QAPP (TtNUS, 2004).
Laboratory Duplicate	One per 20 environmental samples analyzed for inorganic target analytes.	See Table 7-1 of TtNUS (2004)	Laboratory action taken per LTL-1008. TtNUS action taken per validation protocols and Section 11.3 of SWMUs 8 and 15 QAPP (TtNUS, 2004).
Laboratory Method Blank	One per 20 environmental samples or per preparation batch, whichever is more frequent.	< RL (soil and water)	Laboratory action taken per LTL-1008. TtNUS action taken per validation protocols and Section 11.3 of SWMUs 8 and 15 QAPP (TtNUS, 2004).
Matrix Spike ⁽¹⁾	One per analytical fraction per 20 environmental samples for organic target analytes	See Table 7-2 of TtNUS (2004)	Laboratory action taken per LTL-1008. TtNUS action taken per validation protocols and Section 11.3 of SWMUs 8 and 15 QAPP (TtNUS, 2004).
Matrix Spike Duplicate ⁽¹⁾	One per analytical fraction per 20 environmental samples analyzed for organic target analytes.	See Table 7-1 of TtNUS (2004)	Laboratory action taken per LTL-1008. TtNUS action taken per validation protocols and Section 11.3 of SWMUs 8 and 15 QAPP (TtNUS, 2004).

TABLE 4-1

**NON-CALIBRATION QC SAMPLE USAGE FREQUENCIES,
ACCEPTANCE LIMITS, AND CORRECTIVE ACTIONS
UXO 5 AND UXO 7 QAPP ADDENDUM NO. 2**

**NSWC CRANE
CRANE, INDIANA
PAGE 2 OF 2**

QC Sample Type	Collection Frequency	Acceptance Limits	Corrective Action
Surrogate	At least one per sample for organic chromatographic analyses (GC, GC/MS, and HPLC).	See Table 7-2 of TtNUS (2004)	Laboratory action taken per LTL-1008. TtNUS action taken per validation protocols and Section 11.3 of SWMUs 8 and 15 QAPP (TtNUS, 2004).
Temperature Blank	One blank per sample cooler.	$4 \pm 2^{\circ}\text{C}$	Laboratory action taken per LTL-1008. TtNUS action taken per validation protocols and Section 11.3 of SWMUs 8 and 15 QAPP (TtNUS, 2004).

- 1 Matrix spikes and matrix spike duplicates are not analyzed in the field, but additional sample material must be collected in the field to ensure that the laboratory has enough material for spiking and duplicate analysis.

GC/MS - Gas chromatography/mass spectrometry.

$^{\circ}\text{C}$ - Degrees Celsius.

HPLC - High-performance liquid chromatography.

NA - Not applicable.

RL - Reporting limit.

RPD Relative percent difference.

5.0 REFERENCES

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USACE WES (United States Army Corps of Engineers Waterways Experiment Station), 1991. Draft Report, RCRA Facility Investigation, Phase II Soils, Old Rifle Range for: SWMU 07/09, Naval Surface Warfare Center, Crane, Indiana. U.S. Army Corps of Engineers, Waterways Experiment Station, Vicksburg, MS. April.

APPENDIX A

FIELD STANDARD OPERATING PROCEDURES

APPENDIX A

FIELD SOP TABLE OF CONTENTS

CTO0034-01	Sample Labeling
CTO0034-02	Sample Identification Nomenclature
CTO0034-03	Sample Custody and Documentation of Field Activities
CTO0034-04	Decontamination of Field Sampling Equipment
CTO0034-05	Soil and Sediment Sampling
CTO0034-06	Sample Preservation, Packaging, and Shipping
CTO0034-07	Field-Portable X-Ray Fluorescence of soil and Sediment Samples
CTO0034-08	Hand Augering
CTO0034-09	Management of Investigation-Derived Waste

STANDARD OPERATING PROCEDURE NUMBER CTO0034-01

SAMPLE LABELING

1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the procedures to be used for labeling sample containers. Sample labels are used to document the sample ID, date, time, analysis to be performed, preservative, matrix, sampler, and the analytical laboratory. A sample label will be attached to each sample container. The label for each container will contain identical information.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Writing utensil

Disposable medical-grade gloves (e.g. latex, nitrile)

Sample logsheets

Required sample containers: All sample containers for analysis by fix-based laboratories will be supplied and deemed certified clean by the laboratory.

Preprinted sample labels

Chain-of-custody records

Sealable polyethylene bags

Heavy-duty cooler

Ice

3.0 PROCEDURES

3.1 The following information will be electronically printed on each sample label during field activities.

- Project number (CTO 00034)
- Project location (NSWC Crane)
- Sample ID
- Preservative
- Analysis to be performed

- Matrix type
 - Laboratory name
- 3.2 Fill in appropriate information on the electronic Sample Collection Log Form which will automatically update the Chain-of-Custody Form upon submitting the information.
- 3.3 Select the containers that are appropriate for a given sample. Print the associated sample label and affix to the sample container.
- 3.4 Fill the appropriate containers with sample material. Securely close the container lids without overtightening.
- 3.5 Check to determine if the information printed on the label is correct.
- 3.6 Place the sample container in a ziplock plastic bag and place in a cooler containing ice.

Example of a sample label is attached at the end of this SOP.

4.0 ATTACHMENTS

1. Sample Label

ATTACHMENT 1 SAMPLE LABEL

Tetra Tech NUS, Inc. 661 Andersen Drive Pittsburgh, 15220 (412)921-7090		Project:
		Location:
		CTO:
Sample No:		Matrix:
Date:	Time:	Preserve:
Analysis:		
Sampled by:		Laboratory

STANDARD OPERATING PROCEDURE NUMBER CTO0034-02

SAMPLE IDENTIFICATION NOMENCLATURE

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to establish a consistent sample nomenclature system that will facilitate subsequent data management at the Naval Surface Warfare Center (NSWC). The sample nomenclature system has been devised such that the following objectives can be attained.

- Sorting of data by site, location, or matrix
- Maintenance of consistency (field, laboratory, and database sample numbers)
- Accommodation of all project-specific requirements
- Accommodation of laboratory sample number length constraints
- Ease of sample identification

The NSWC Crane Environmental Protection Department must approve any deviations from this procedure.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Pen with indelible ink

Sample tags

Sample container labels

3.0 SAMPLE IDENTIFICATION NOMENCLATURE

3.1 Confirmation Samples

All confirmation samples will be properly labeled with a sample label affixed to the sample container. Each sample will be assigned a unique sample tracking number.

3.1.1 Confirmation Sample numbering Scheme

The sample tracking number will consist of a four- or five-segment alpha-numeric code that identifies the sample's associated Unexploded Ordnance (UXO) site, sample type, location, and for aqueous samples, where applicable, whether a sample is filtered, and/or the sample round number. For soil and sediment samples, the final four tracking numbers will identify the depth in units of feet below ground surface at which the sample was collected.

The alphanumeric coding to be used is explained in the following diagram and subsequent definitions:

AN	AA	NNNA	NNNN (Soils and Sediment only)
UXO Number	Matrix	Sample Location Number and Grab or Composite	Sequential depth interval from freshly exposed surface

Character Type:

A = Alpha
 N = Numeric

UXO Number (AN):

X7 = UXO 7
 X5 = UXO 5

Matrix Code (AA):

SS = Surface Soil Sample

Individual aliquots used to form composite samples will be designated "A", "B", "C", "D", "etc.". Each of the aliquots for a given sample will be recorded in the field log. The number of aliquots combined to form a composite and the locations of the aliquots within the sampled area must be recorded in the "OBSERVATIONS/NOTES" section of the Soil and Sediment Sample Log Sheet.

Location Number (NNNA):

Sequential number beginning with "001" for each matrix, followed by a letter indicating grab (G) or composite (C) sample.

Depth Interval:

This code section will be used for soil and sediment samples only.

The depth code is used to note the depth below ground surface (bgs) at which a soil or sediment sample is collected. The first two numbers of the four-number code specify the top interval, and the third and fourth specify the bottom interval in feet bgs of the sample. The depths will be noted in whole numbers only; further detail, if needed, will be recorded on the sample log sheet, boring log, logbook, etc. In certain cases, where a sample may be composite sample, "location" refers to a particular sampling grid represented by a composite sample. Each composite sample will be comprised of aliquots identified as "A", "B", "C", etc. used to identify individual aliquots associated with a particular sample.

3.1.2 Examples of Confirmation Sample Nomenclature

The first composite surface soil sample collected from UXO 5, sampling location 003, at a depth of 2 feet would be labeled as "X5SS001C0002". The individual aliquots making up the composite sample should be described as Aliquots "A", "B", "C", etc. for that particular sample and noted on the sample log sheet.

3.3 Field Quality Assurance/Quality Control (QA/QC) Sample Nomenclature

Field QA/QC samples are described in the QAPP Addendum No. 1 (TINUS, 2006) and this QAPP Addendum No. 2. They will be designated using a different coding system than the one used for regular field samples.

3.3.1 QC Sample Numbering

The QC code will consist of a three- to four-segment alpha-numeric code that identifies the sample QC type, the date the sample was collected, and the number of this type of QC sample collected on that date.

AA	NNNNNN	NN
QC Type	Date	Sequence Number (per day)

The QC types are identified as:

TB = Trip Blank

FD = Field Duplicate

The sampling time recorded on the Chain-of-Custody Form, labels, and tags for duplicate samples will be 0000 so that the samples are "blind" to the laboratory. Notes detailing the sample number, time, date, and type will be recorded on the sample log sheets and will document the location of the duplicate sample (sample log sheets are not provided to the laboratory).

3.3.2 Examples of Field QA/QC Sample Nomenclature

The first duplicate of the day at UXO 5 for a surface soil sample collected on March 24, 2007 would be designated as X5FD03240701.

The third duplicate of the day taken at UXO 7 of a surface soil sample collected on April 12, 2007 would be designated as X7FD04120703.

The first trip blank associated with samples collected on October 12, 2007 would be designated as TB10120701.

STANDARD OPERATING PROCEDURE NUMBER CT034-03

SAMPLE CUSTODY AND DOCUMENTATION OF FIELD ACTIVITIES

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedures for sample custody and documentation of field sampling and field analyses activities.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

The following logbooks, forms, and labels are required.

Site logbook

Field logbook

Sample label

Chain-of-Custody Form

Custody seals

Equipment calibration log

Monitoring Well Inspection Form

Water-Level Measurement Form

Low-Flow Purge Data Sheet

Soil and Sediment Sample Log Sheet

Ground Water Sample Log Sheet

Surface Water Sample Log Sheet

3.0 PROCEDURES

This section describes custody and documentation procedures. All entries made into the logbooks, custody documents, logs, and log sheets described in this SOP must be made in indelible ink (black is preferred). No erasures are permitted. If an incorrect entry is made, the entry will be crossed out with a single strike mark, initialed, and dated. Note: In lieu of the above logs, electronic documentation may be used to store data.

3.1 Site Logbook

The site logbook is a hard-bound, paginated, controlled-distribution record book in which all major on-site activities are documented. At a minimum, the following activities and events will be recorded (daily) in the site logbook:

- All field personnel present
- Arrival/departure of site visitors
- Arrival/departure of equipment
- Start or completion of sampling activities
- Daily on-site activities performed each day
- Sample pickup information
- Health and safety issues
- Weather conditions

The site logbook is initiated at the start of the first on-site activity (e.g., site visit or initial reconnaissance survey). Entries are to be made for every day that on-site activities take place.

The following information must be recorded on the cover of each site logbook:

- Project name
- Project number
- Book number
- Start date
- End date

Information recorded daily in the site logbook need not be duplicated in other field notebooks but must summarize the contents of these other notebooks and refer to specific page locations in these notebooks for detailed information (where applicable). At the completion of each day's entries, the site logbook must be signed and dated by the field operations leader (FOL).

3.2 Field Logbooks

The field logbook is a separate dedicated notebook used by field personnel to document his or her activities in the field. This notebook is hardbound and paginated.

3.3 Sample Labels

Adhesive sample container labels must be completed and applied to every sample container. Information on the label includes the project name, location, sample number, date, time, preservative, analysis, matrix, sampler's initials, and the name of the laboratory performing the analysis.

3.4 Chain-of-Custody Form

The Chain-of-Custody Form (COC) is a multi-part form that is initiated as samples are acquired and accompanies a sample (or group of samples) as it is transferred from person to person. Each COC is numbered. This form must accompany any samples collected for laboratory chemical analysis. A copy of a blank COC form is attached at the end of this SOP.

The FOL or must include the name of the laboratory in the "Remarks" section to ensure that the samples are forwarded to the correct location. If more than one COC is necessary for any cooler, the FOL will indicate "Page ___ of ___" on each COC. The original (top) signed copy of the COC will be placed inside a large Ziploc-type bag and taped inside the lid of the shipping cooler. Once the samples are received at the laboratory, the sample custodian checks the contents of the cooler(s) against the enclosed COC(s). Any problems are noted on the enclosed COC Form (bottle breakage, discrepancies between the sample labels, COC form, etc.) and will be resolved through communication between the laboratory point-of-contact and the Task Order Manager (TOM). The COC form is signed and retained by the laboratory and becomes part of the sample's corresponding analytical data package.

3.5 Custody Seal

The custody seal is an adhesive-backed label with a number on each seal. It is part of the chain-of-custody process and is used to prevent tampering with samples after they have been collected in the field and sealed in coolers for transit to the laboratory. The custody seals are signed and dated by the samplers and affixed across the opening edges of each cooler (two seals per cooler) containing environmental samples. The laboratory sample custodian will examine the custody seal for evidence of tampering and will notify the TtNUS TOM if evidence of tampering is observed.

3.6 Equipment Calibration Log

The Equipment Calibration Log is used to document calibration of measuring equipment used in the field. The Equipment Calibration Log documents that the manufacturer's instructions were followed for calibration of the equipment, including frequency and type of standard or calibration device. An Equipment Calibration Log must be maintained for each electronic measuring device requiring calibration. Entries must be made for each day the equipment is used.

3.12 Soil and Sediment Sample Log Sheet

The Soil and Sediment Sample Log Sheets are used to document the sampling of soils and sediments (see SOP CTO0034-05).

4.0 ATTACHMENTS

1. Chain-of-Custody Record

3/99
FORM NO. TtNUS-001

NSWC Crane
QAPP Addendum No.2
Revision: 0
Date: March 2007
Section: SOP_CTO0034-03
Page 5 of 5

STANDARD OPERATING PROCEDURE NUMBER CTO0034-04

DECONTAMINATION OF FIELD SAMPLING EQUIPMENT

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedures to be followed when decontaminating non-dedicated field sampling equipment during the field investigations at the NSWC Crane facility.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Waterproof pens

Non-latex rubber or plastic gloves

Cotton gloves

Field logbook

Potable water

Deionized water

LiquiNox detergent

Brushes, spray bottles, paper towels, etc.

55-gallon drum or other container to collect and transport decontamination fluids

3.0 DECONTAMINATION PROCEDURES

- 3.1 Don non-latex and/or cotton gloves and decontaminate sampling equipment (in accordance with the following steps) prior to field sampling and between samples.
- 3.2 Rinse the equipment with potable water. Rinsing may be conducted by spraying with water from a spray bottle or by dipping. Collect the potable water rinsate into a container.
- 3.3 Wash the equipment with a solution of LiquiNox detergent. Prepare the LiquiNox wash solution in accordance with the instructions on the LiquiNox container. Collect the LiquiNox wash solution into a container. Use brushes or sprays as appropriate for the equipment. If oily residue has

accumulated on the sampling equipment, remove the residue with an isopropanol wash and repeat the Liquinox wash.

- 3.4 Rinse the equipment with potable water. Rinsing may be conducted by spraying with water from a spray bottle or by dipping. Collect the potable water rinsate into a container.
- 3.5 Rinse the equipment with deionized water. Rinsing may be conducted by spraying with water from a spray bottle or by dipping. Collect the deionized water rinsate into a container.
- 3.6 Remove excess water by air drying, shaking, or by wiping with paper towels as necessary.
- 3.7 Document decontamination by recording it in the field logbook.
- 3.8 Containerized decontamination solutions will be managed in accordance with the procedures described in SOP CTO0034-09 and the QAPP Addendum No. 2.

STANDARD OPERATING PROCEDURE NUMBER CTO0034-05

SOIL AND SEDIMENT SAMPLING

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedure for collecting surface soil samples using the hand auger technique to support RCRA Facility Assessments at UXO 5 and UXO 7 in accordance with the requirements of this QAPP Addendum No. 2.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

The following field forms and equipment are required for sediment sampling.

Disposable medical-grade gloves (e.g., latex, nitrile)

Indelible marker

Stainless steel auger buckets

Stainless steel extension rods

Cross handle

Plastic storage bags

Sample tags

Shipping containers (containing ice)

Disposable plastic trowels or Stainless steel trowels

Stainless steel mixing bowls

Sample containers: Sample containers are certified clean by the laboratory supplying the containers.

Electronic notebook containing:

Soil Sample Log Forms.

Daily Activity Logs.

Chain-of-Custody Form.

Soil Boring Log.

Electronic label maker

3.0 SAMPLING PROCEDURES

Hand augering will be employed to collect soil cores. The sampler will wear clean, disposable, medical-grade gloves.

- 3.1 Clear the area to be sampled of any surface debris (herbaceous vegetation, twigs, rocks, litter, etc.).
- 3.2 Turn the hand auger sampler into the ground to a depth of 2 feet. The 0- to 2-foot depth soil interval is considered to be the surface soil.
- 3.3 Log the soil core on the Boring Log Sheet (see SOP CTO034-07).
- 3.4 For a grab sample, place the soil core in a stainless-steel mixing bowl and homogenize. For a composite sample, place equal amounts of the soil cores in a stainless-steel mixing bowl and homogenize. Place soil sample into laboratory supplied sampling container, print, and affix electronically printed label.
- 3.5 For samples that are to be submitted for laboratory analysis place the labeled sample container into a plastic storage bag and then place the plastic storage bag holding the sample container into a cooler containing ice.
- 3.6 Complete required information on the electronic Soil Sample Log Sheet (copy attached at the end of this SOP). Note the location of each aliquot in the "OBSERVATIONS/NOTES" section of the Soil and Sediment Sample Log Sheet. The information will be automatically updated to the Chain-of-Custody (COC) Form.
- 3.7 For field XRF analysis, remove a portion of the sample from the container. Retain the remainder of the sample for submittal for laboratory analysis, as appropriate.
- 3.8 Record the date, sampling site, site conditions, location map, and other information on the Soil and Sediment Sample Log Sheet.

4.0 ATTACHMENTS

1. Soil and Sediment Sample Log Sheet

ATTACHMENT 1
SOIL AND SEDIMENT SAMPLE LOG SHEET

SOIL & SEDIMENT SAMPLE LOG SHEET

Page ____ of ____

Project Site Name: _____ Project No.: _____ <input type="checkbox"/> Surface Soil <input type="checkbox"/> Subsurface Soil <input type="checkbox"/> Sediment <input type="checkbox"/> Other: _____ <input type="checkbox"/> QA Sample Type: _____		Sample ID No.: _____ Sample Location: _____ Sampled By: _____ C.O.C. No.: _____ Type of Sample: <input type="checkbox"/> Low Concentration <input type="checkbox"/> High Concentration	
---	--	--	--

GRAB SAMPLE DATA:			
Date:	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Time:			
Method:			
Monitor Reading (ppm):			

COMPOSITE SAMPLE DATA:				
Date:	Time	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Method:				
Monitor Readings (Range in ppm):				

SAMPLE COLLECTION INFORMATION:			
Analysis	Container Requirements	Collected	Other

OBSERVATIONS / NOTES:		MAP:

Circle if Applicable:		Signature(s):
MS/MSD	Duplicate ID No.:	

STANDARD OPERATING PROCEDURE NUMBER CTO034-06

SAMPLE PRESERVATION, PACKAGING, AND SHIPPING

1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the procedures for sample preservation, packaging, and shipping to be used in handling aqueous samples, soils, and sediments collected at the NSWC Crane.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Shipping labels

Custody seals

Chain-of-custody (COC) form(s)

Sample containers with preservatives: All sample containers for analysis by fixed-base laboratories will be supplied, with preservatives added (if required) and deemed certified clean by the laboratory.

Sample shipping containers (coolers): All sample shipping containers are supplied by the laboratory.

Packaging material: Bubble wrap, ZipLoc[®] bags, strapping tape, etc.

3.0 PROCEDURES FOR SAMPLE PRESERVATION, PACKAGING, AND SHIPPING

- 3.1 The laboratory provides sample containers with preservative already included (as required) for the analytical parameter for which the sample is to be analyzed. All samples will be held, stored, and shipped at 4°C. This will be accomplished through refrigeration (used to hold samples prior to shipment) and/or ice.
- 3.2 The sampler shall maintain custody of the samples until the samples are relinquished to another custodian or to the common carrier.
- 3.3 Check that each sample container is properly labeled, the container lid is securely fastened, and the container is sealed in a ZipLoc[®] bag.

- 3.4 If the container is glass, place the sample container into a bubble-out shipping bag and seal the bag using the self-sealing, pressure sensitive tape supplied with the bag.
- 3.5 Inspect the insulated shipping cooler. Check for any cracks, holes, broken handles, etc. If the cooler has a drain plug, make certain it is sealed shut, both inside and outside of the cooler. If the cooler is questionable for shipping, the cooler must be discarded.
- 3.6 Put ice into ZipLoc[®] bags and place a layer of the sealed bags on the bottom of the cooler. Place the sample containers into the shipping cooler on top of the ice in an upright position (containers will be upright, with the exception of the 40-ml vials). Place ZipLoc[®] bags of ice flat against the sides of the cooler. Continue filling the cooler with samples until the cooler is nearly full and the movement of the sample containers is limited.
- 3.7 Place a temperature blank in the cooler. Make sure that all samples identified for VOC analysis and their associated trip blank are packed in the same cooler (i.e., VOC samples and trip blank that were stored together during sampling activities can not be separated into different coolers for shipping purposes).
- 3.8 Add a final layer of ice sealed in ZipLoc[®] bags to the top of the samples just before the cooler is closed and sealed.
- 3.9 Place the original (top) signed copy of the COC form inside a large ZipLoc[®] bag. Tape the bag to the inside of the lid of the shipping cooler that contains the samples for VOC analysis.
- 3.10 Close the cooler and seal the cooler with approximately four wraps of strapping tape at each end of the cooler. Prior to wrapping the last wrap of strapping tape, apply a signed, numbered, and dated custody seal to each side of the cooler (one per side). Cover the custody seal with the last wrap of tape. This will provide a tamper evident custody seal system for the sample shipment.
- 3.11 Affix a shipping label to the top of the cooler containing samples for VOC analysis, ensuring all of the shipping information is filled in properly. Overnight (e.g., FedEx Priority Overnight) courier services will be used for all sample shipments.

- 3.12 All samples will be shipped to the laboratory no more than 72 hours after collection. Under no circumstances will sample hold times be exceeded.

STANDARD OPERATING PROCEDURE NUMBER CTO0034-07

FIELD PORTABLE X-RAY FLUORESCENCE ANALYSIS OF SOIL AND SEDIMENT SAMPLES USING THE INNOV-X ALPHA SERIES INSTRUMENT

1.0 PURPOSE

This procedure is for the semiquantitative analysis of metallic lead particles and chemical compounds of lead in soil using a field portable x-ray fluorescence (FPXRF) spectrometer. This procedure is based on the United States Environmental Protection Agency (USEPA)-approved XRF field screening method for elemental analysis (Method 6200).

2.0 SCOPE, APPLICATION, AND LIMITATIONS

2.1 Scope of Procedure

Analysis of any other elements beside lead using FPXRF may require changes to this Standard Operating Procedure (SOP), and are therefore outside the scope of this SOP.

Although it is possible to use FPXRF to measure analytes in situ, this SOP requires removal of a soil sample from its native environment prior to analysis. By removing, drying, and homogenizing the sample prior to analysis, more precise and accurate results are obtained.

2.2 Analyst Training

Use of this method is restricted to personnel both trained and knowledgeable in the operation of the Innov-X alpha series XRF instrument or under the supervision of a trained and knowledgeable individual. Proper training for the safe operation of the instrument should be completed by the analyst prior to analysis. This training may be obtained directly from INNOV-X, an INNOV-X instrument distributor or lessor, or another trained Tetra Tech NUS, Inc. person.

3.0 ACRONYMS AND ABBREVIATIONS

FPXRF: Field portable x-ray fluorescence.

mg/kg: milligrams per kilogram.

MDL: Method detection limit.

PQL: Practical quantitation limit.

QC: Quality control.

RPD: Relative percent difference.

USGS: United States Geological Survey.

XRF: X-ray fluorescence.

4.0 RESPONSIBILITIES

Analyst/Chemist - Responsible for all aspects of sample preparation and analysis including equipment maintenance. Also responsible for maintaining chain-of-custody of samples after receipt from sampling personnel.

5.0 PROCEDURES

5.1 Safety

5.1.1 Radiation Safety

Radiation safety practices for the INNOV-X instrument can be found in the operator's manual. Protective shielding should never be removed by the analyst or any personnel other than the manufacturer.

An additional hazard present with x-ray tubes is the danger of electric shock from the high voltage supply. The danger of electric shock is as substantial as the danger from radiation but is often overlooked because of its familiarity.

5.1.2 Protective Equipment

Analysts must wear disposable plastic gloves whenever sample aliquots are being transferred from one vessel to another. Consult the health and safety plan for other protection requirements.

5.2 Apparatus and Materials

Apparatus and materials consist of the following:

INNOV-X Alpha Series FPXRF spectrometer with data processing unit (iPAQ) pocket personnel computer):

INNOV-X Alpha Series XRF instrument manual to match the INNOV-X Alpha Series instrument.

Aluminum drying pans or aluminum foil: Sized suitably to hold as much as 50 grams of sample and fit into the drying oven.

Calibration verification check sample: A National Institute of Standards and Technology (NIST) or other Standard reference material (SRM) that contains lead in a concentration range that is compatible with the project objectives to verify the accuracy of the instrument. SRMs can be obtained from the NIST, the U.S. Geological Survey (USGS), the Canadian National Research Council, and the national bureau of standards in foreign nations. Pertinent NIST SRMs for FPXRF analysis include 2704, Buffalo River Sediment; 2709, San Joaquin Soil; and 2710 and 2711, Montana Soil. These SRMs contain soil or sediment from actual sites that has been analyzed using independent inorganic analytical methods by many different laboratories. Acceptable limits for SRM percent recoveries are usually provided with the SRM. In their absence, a limit of ± 30 percent will be used as a guideline.

Instrument Blank: May be silicon dioxide, a Teflon block, a quartz block, "clean" sand, or lithium carbonate.

Lead calibration check standard: Supplied by the FPXRF manufacturer.

Method blank material for performing method blank checks: May be lead-free silica sand or lithium carbonate that undergoes the same preparation procedure as the samples.

Battery charger.

Polyethylene sample cups: 31 millimeters (mm) to 40 mm in diameter with collar, or equivalent (appropriate for FPXRF instrument).

X-ray window film: Mylar™, Kapton™, Spectrolene™, polypropylene, or equivalent; 2.5 to 6.0 micrometers (μm) thick.

Sample containers: glass or plastic to store samples.

Sieves: 60-mesh Stainless steel, Nylon, or equivalent for preparing soil and sediment samples if necessary.

Trowels: for collecting soil samples.

Plastic bags: used for collection and homogenization of soil samples. May also be used as sample presentation device.

Drying oven: standard convection or toaster oven, for soil samples that require drying.

Rolling pin (optional): Wooden rolling pin for crushing samples.

5.3 Sample Collection, Preservation, and Handling

Samples shall be provided to the FPXRF analyst in plastic bags. The analyst is responsible for maintaining chain-of-custody of all samples until all analyses have been successfully completed. No sample preservation is necessary. All samples shall be handled in accordance with sample handling SOPs in effect for the field event.

5.4 Preventive Maintenance

Refer to the instrument manual for specific manufacturer's recommendations.

5.5 Instrument Start-Up

- 5.5.1 Ensure the pocket PC (iPAQ) is plugged into the FPXRF instrument body and install a fully charged battery into the instrument.
- 5.5.2 Press the ON/OFF button on the base of the pistol grip of the instrument. If the iPAQ does not automatically power up, press the Power button in the top right corner of the iPAQ.
- 5.5.3 Tap the Microsoft icon at the upper left corner of the iPAQ.
- 5.5.4 Chose START.

5.5.5 Tap "Soil Mode" on the menu or choose Mode (bottom of screen) and then choose Soil Mode from the drop down menu.

5.5.6 Allow the instrument to warm up (approximately 3 minutes).

5.5.7 Release the manual trigger lock.

5.5.8 Standardize the instrument in accordance with Section 5.6.

5.6 Standardization/Calibration Check

It is not possible to start an analysis if the instrument has not been standardized. To verify proper calibration of the instrument it is necessary to periodically standardize it using the automated standardization procedure. This must be done anytime the instrument is restarted and every 4 hours of operation, although re-standardization may be done at any other time (e.g., when instrument drift is suspected).

5.6.1 Click the standardization piece (supplied with the instrument) on the front of the instrument, verifying that the solid portion of the standard completely covers the analysis window.

5.6.2 Select "Tap here to Standardize" or select *File* → *Standardize*. The red light on top of the instrument will blink indicating that the instrument is producing x-rays and the shutter is open. The amber light on the rear of the instrument will also be illuminated and a status bar will appear to display the progress of the standardization.

5.6.3 Upon successful standardization the message "Successful Standardization" will appear along with the instrument resolution. In this case tap "ok" to dismiss the completion message. If problems are encountered, either follow the prompts that appear and/or repeat the standardization. Contact the FOL if problems persist. Take note of any error messages that appear as they may be useful if the instrument manufacturer must be contacted. Additional assistance is also available in the manufacturer's instrument manual.

5.7 Quality Control

The quality control (QC) program includes analysis of blanks calibration verification checks, duplicate analyses, and field duplicate samples. For all the above areas, any identified problems and corrective

action must be documented in the instrument run log, analysis narrative report, and instrument maintenance log or standards log (as applicable). Identical operating conditions will be used for each sample.

5.7.1 Laboratory Blanks

Two types of blank samples shall be analyzed for FPXRF analysis: instrument blanks and method blanks.

5.7.1.1 At the beginning of each day, at the end of each day, and after every 20th sample or when potential contamination of the instrument is suspected, analyze an instrument blank to verify that no contamination exists in the spectrometer or on the probe window.

If the lead concentration in the blank exceeds the method detection limit (MDL), see Section 5.9.3) check the probe window and other potentially contaminated instrument components for contamination. If contamination is not causing the elevated blank readings, "zero" the instrument according to manufacturer's instructions.

5.7.1.2 After every 20th sample analyze a method blank. If the method blank lead concentration exceeds the practical quantitation limit (PQL, see Section 5.9.4), identify the cause of the elevated lead concentration and reanalyze all samples since the last acceptable method blank.

5.7.2 Calibration Verification Checks

5.7.2.1 After performing each blank check (Section 5.7.2), analyze a calibration verification check sample to check the accuracy of the instrument and to assess the stability and consistency of the analysis for the analytes of interest.

5.7.2.2 If the measured lead percent recovery (See Section 5.9.1) is less than 60 percent or greater than 135 percent, reanalyze the check sample. If the value continues to fall outside this acceptance range, the instrument should be recalibrated, or restandardized according to the manufacture instructions and the batch of samples analyzed before the unacceptable calibration verification check must be reanalyzed.

5.7.3 XRF Duplicate Samples

XRF duplicate samples are two portions of the same sample that have been prepared and homogenized together, and then split and analyzed in the same manner by the XRF analyst.

5.7.3.1 Analyze an XRF duplicate at a frequency of 1 per 20 or once per day, whichever is more frequent.

5.7.3.2 If the computed RPD (See Section 5.9.2) exceeds 50 percent reanalyze both samples. If the RPD again exceeds 50 percent RPD consider whether the high degree of imprecision is caused by sample heterogeneity or other causes. This assessment may be aided by repeating the analysis of a sample that was analyzed previously. If the observed imprecision is attributed to sample heterogeneity, increase the number of readings made per sample to try to limit the imprecision and repeat the analyses. If this does not correct the problem notify the FOL.

5.8 Sample Analysis

Note:

This section provides sample analysis instructions, assuming that appropriate instrument start-up and calibration checks have been completed. The longer the instrument count time, the lower the detection limits and the less uncertainty there is with a recorded result. Count time is user-selectable through the instrument's software. Because the XRF data will be used in a screening capacity to make preliminary decisions concerning the soil concentrations relative to 400 mg/kg, it is not necessary to obtain a high degree of accuracy or precision with the instrument. Therefore, count times should be limited to less than 180 seconds unless an unusually high degree of precision is expected. To change the count time, select Options → Setup Testing and enter the same value (in seconds) to minimum and maximum count times.

Note:

Section 5.7 identifies the appropriate frequencies for conducting various QC sample analyses and the associated acceptance limits and corrective actions for potentially unsuitable conditions. The specified QC analysis frequencies are minimum frequencies. More frequent QC sample analyses are permitted, especially when diagnosing quality problems.

5.8.1 Ensure that calibration checks and blanks have been analyzed according to Sections 5.6, 5.7.1, and 5.7.2. Count times shall be at least 60 seconds but generally less than 180 seconds.

5.8.2 Acquire enough soil sample to fill an 8-ounce jar and separate from it all particles greater than the size of a pea.

- 5.8.3 Homogenize the remaining finer grained portion of the sample by simple mixing until it appears as uniform in texture and composition as practicable. Mixing may be done in a beaker or other suitable lead-free container. If the sample is moist and has high clay content, it may be kneaded in a plastic bag. Mixing shall continue for at least two minutes to ensure that the sample is well mixed. To aid mixing, the sample may be placed into a thick-walled (3 mil or thicker) gallon-sized freezer bag (e.g., ZipLoc[®]) and rolled flat with a rolling pin to break up large chunks of dirt.
- 5.8.4 Place approximately 20 to 50 grams (one U.S. nickel weighs about 5 grams) in a suitable container (e.g., aluminum drying pan) for drying.
- 5.8.5 Dry the homogenized sample from Step 5.8.4 for approximately 20 to 30 minutes in the oven at a temperature not greater than 150°C (a setting of approximately 300 F). If the sample is not visibly dry after this initial drying time, place the sample back into the oven until the sample is dry.
- 5.8.6 Re-homogenize the dried sample aliquot in a beaker or other suitable lead-free container to obtain a well mixed soil sample. Mixing shall continue for at least one minute.
- 5.8.7 Place a portion (approximately 1.5 cubic inches) of the dried, homogenized sample aliquot into the instrument manufacturer's recommended sample cup (e.g., a 31.0-mm polyethylene sample cup (or equivalent) or place it in a thin-walled (1.0 mil or thinner) plastic sandwich bag (e.g., ZipLoc[®]).
- 5.8.8 If using a disposable plastic sample cup, ensure the cup is at least three-quarters full and cover with mylar (or other) film per the manufacturer's recommendations.
- 5.8.9 Present the sample to the instrument in Soil Mode.
- 5.8.10 Perform a single pull of the trigger to start the count. The count time shall be the same as was used for the calibrations, calibration checks, and blank analyses. The message "Test in progress" will appear on the instrument and the red light on top of the instrument and will illuminate.
- 5.8.11 When the predetermined count period has expired the message "Test complete" will appear on the instrument. A slight delay may also be incurred during which time the message "calculating" may appear to indicate that results are being computed.

5.8.12 Record the displayed results for lead concentration in mg/kg and the associated error on Figure 1.

CAUTION

Inconsistent positioning of samples in front of the probe window is a potential source of error because the x-ray signal decreases by the square of the distance from the radioactive source. This error is minimized by maintaining the same distance between the window and each sample. For the best results, the window of the probe should be in direct contact with the sample, which means that the sample surface should be flat and smooth to provide a good contact surface.

5.8.13 Remix the sample in the plastic bag (or rotate the sample cup approximately one-third of a turn) then acquire another measurement by repeating Steps 5.8.10 and 5.8.11. Record the result and associated error on Figure 1.

5.8.14 Repeat Steps 5.8.10 and 5.8.11. Record the result and associated error on Figure 1 (XRF Field Form).

5.8.15 Based on the degree of precision demonstrated by the three individual measurements, determine whether additional readings should be acquired on the sample. This determination shall be based on professional judgment of the FPXRF analyst and shall consider the degree of precision observed during calibration checks and previous sample analyses. The objective will be to ensure that the average reading reported for each sample is representative of the true sample concentration. If the analyst feels that non-representative readings are being obtained the analyst shall correct the analytical system or notify the FOL prior to continuing with analyses.

5.8.16 Ensure that measured results are reported to the following standards

- Results < 1000 mg/kg (or parts per million) are reported to two significant figures and results > 1000 mg/Kg are reported to three significant figures.
- All values < MDL shall be reported as the MDL and flagged with the letter "U".
- All values > MDL and < PQL shall be reported as is and flagged with the letter "B".

5.9 Calculations

5.9.1 Percent Recovery: The equation for determining percent recovery of calibration verification check standards and standard reference materials is:

$$\%R = \frac{\text{Experimental Concentration}}{\text{Certified or Known Concentration}} \times 100 \%$$

5.9.2 Relative Percent Difference: The equation for determining relative percent difference for laboratory and field duplicate samples is:

$$RPD = \frac{|\text{Amount in Sample 1} - \text{Amount in Sample 2}|}{0.5 (\text{Amount in Sample 1} + \text{Amount in Sample 2})} \times 100 \%$$

5.9.3 Method Detection Limit (MDL): Because the analyses governed by this SOP are semi-quantitative, the manufacturer-specified detection limit will be reported as the MDL unless the specified detection limit is less than 20 mg/kg. Care will be taken to ensure that the appropriate count time is consistent with the reported detection limit. However, no value less than 20 mg/kg will be reported as an MDL.

5.9.4 Practical Quantitation Limit (PQL): Multiply the MDL by 3 to obtain the PQL: $PQL = MDL \times 3$

6.0 REFERENCES

Innov-X Systems, Inc. Innov-X Systems X-Ray Fluorescence Spectrometers Instruction Manual. Woburn, MA. June 2002.

Stephen Shefsky, NITON Corporation. Comparing Field Portable X-Ray Fluorescence (XRF) To Laboratory Analysis of Heavy Metals In Soil. Presented at the International Symposium of Field Screening Methods for Hazardous Wastes and Toxic Chemicals. Las Vegas, Nevada. January 29-31, 1997.

USEPA (U.S. Environmental Protection Agency), Method 6200: Field Portable X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment, Office of Solid Waste, Washington, D.C. January 1998.

USEPA, Region I, Northeast Waste Management Officials' Association (NEWMOA) Technology Review Committee Advisory Opinion. Innovative Technology: X-Ray Fluorescence Field Analysis. September 21, 1999.

FIGURE 1

FIELD PORTABLE XRF SAMPLE ANALYSIS LOG SHEET

Page ____ of ____

Project Site Name: _____				Run Length/Duration: _____							
Project No.: _____				Measurement Units: _____							
Analyzed By: _____				Reference/Calibration: _____							
SAMPLING AND FIELD PORTABLE XRF RESULTS DATA:											
SAMPLE ID	ANALYSIS DATE	ANALYSIS TIME	ELEMENT ANALYSIS	RESULT RUN 1	RESULT RUN 2	RESULT RUN 3	RESULTS AVERAGE	ERROR RUN 1	ERROR RUN 2	ERROR RUN 3	ERROR AVERAGE

STANDARD OPERATING PROCEDURE NUMBER CTO034-08

SOIL CORING USING HAND AUGER TECHNIQUES

1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the procedures for collecting surface soil cores from unconsolidated overburden materials using hand augering techniques at the NSWC Crane facility.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Disposable medical-grade gloves (e.g., latex, nitrile)

Writing utensil

Stainless Steel Auger Buckets

Stainless Steel Extension Rods

Cross Handle

Required decontamination materials

Bentonite pellets

3.0 BOREHOLE ADVANCEMENT AND SOIL SAMPLING USING A HAND AUGER

Hand Augers may be employed to collect the soil cores. A hand augering system generally consists of a variety of all stainless steel bucket bits (i.e. cylinders 6-1/2" long and 2-3/4", 3-1/4", and 4" in diameter), a series of extension rods (available in 2', 3', 4' and 5' lengths), a cross handle. A larger diameter bucket bit is commonly used to bore a hole to the desired sampling depth and then withdrawn. In turn, the larger diameter bit is replaced with a smaller diameter bit, lowered down the hole, and slowly turned into the soil at the completion depth or refusal. The apparatus is then withdrawn and the soil sample collected.

- 3.1 The hand auger can be used in a wide variety of soil conditions. It can be used to sample soil, both from the surface, or to depths in excess of 12 feet. However, the presence of rock layers and the collapse of the borehole normally contribute to its limiting factors.

Attach a properly decontaminated bucket bit into a clean extension rod and further attach the cross handle to the extension rod.

- 3.2 Clear the area to be sampled of any surface debris (vegetation, twigs, rocks, letter, etc.)
- 3.3 Begin augering (periodically removing accumulated soils from the bucket bit) and add additional rod extensions as necessary. Also, note (in a field notebook or on standardized data sheets) and changes in the color, texture or odor of the soil.
- 3.4 After reaching the desired depth, slowly and carefully withdraw the apparatus from the borehole.
- 3.5 Remove the soiled bucket bit from the extension rod extension and replace it with another properly decontaminated bucket bit. The bucket bit used for sampling is commonly smaller in diameter than the bucket employed to initiate the borehole.
- 3.6 Carefully lower the apparatus down the borehole. Care must be taken to avoid scraping the borehole sides.
- 3.7 Slowly turn the apparatus until the bucket bit is advanced approximately 6 inches.
- 3.8 Discard the top of the core (approximately 1"), which represents any loose material collected by the bucket bit before penetrating the sample material.
- 3.9 Utilizing a properly decontaminated stainless steel trowel or disposable trowel, remove the sample material from the bucket bit and place into a properly decontaminated stainless steel mixing bowl and thoroughly homogenize the sample material prior to filling the remaining sample containers, as described in SOP CTO 034-05.
- 3.10 Excess soil core materials will be returned to the hole and tamped. If insufficient soil is available to fill the hole to the ground surface, then bentonite pellets mixed with the soil will be used to backfill the hole.
- 3.11 Decontaminate all soil sampling equipment in accordance with SOP CTO034-04 before collecting the next sample.

STANDARD OPERATING PROCEDURE NUMBER CTO0034-09

MANAGEMENT OF INVESTIGATION-DERIVED WASTE

1.0 PURPOSE

This Standard Operating Procedure (SOP) describes how investigation-derived waste (IDW) will be collected, segregated, classified, and managed during the field investigations at the NSWC Crane facility. The following types of IDW will be generated during this investigation:

- Decontamination solutions
- Personal protective equipment and clothing (PPE)
- Miscellaneous trash and incidental items

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Health and safety equipment (with PPE)

Decontamination equipment

Field logbook and indelible ink pen

Plastic sheeting and/or tarps

55-gallon drums with sealable lids

IDW labels for drums

Wastewater container tanks

Plastic garbage bags

3.0 PROCEDURES

Management of IDW includes the collection, segregation, temporary storage, classification, final disposal, and documentation of the waste-handling activities if necessary.

3.1 Liquid Wastes

Liquid wastes that will be generated during the site activities include decontamination solutions from sampling equipment. These wastes will be collected and transported to a central location at NSWC

Crane (adjacent to a Crane-designated sanitary sewer manhole). The water will be discharged to the sewer by gravity draining.

3.2 Solid Wastes

Solid wastes that may be generated during the site activities include collection of lead shot from surface soil samples. This waste will be containerized and handed over to the NSWC Crane Environmental Department Manager at the conclusion of field activities.

3.3 PPE and Incidental Trash

All PPE wastes and incidental trash materials (e.g., wrapping or packing materials from supply cartons, waste paper) will be decontaminated (if contaminated), double bagged, securely tied shut, and placed in a designated waste receptacle at NSWC Crane.

APPENDIX B

LABORATORY STANDARD OPERATING PROCEDURES

LABORATORY STANDARD OPERATING PROCEDURES (SOPs)

TABLE OF CONTENTS

SOP NUMBER	REV #	QAPP	TITLE
LAUCKS TESTING LABORATORIES			
LTL-1001	7	034	Elements of SOP and Method Formats
LTL-1002	8	034	Document Tracking and Control
LTL-1003	4	034	Chain-of-Custody and Documentation Procedures
LTL-1004	6	034	Documentation of Analyst Competence and Training
LTL-1005	6	034	Analytical Balances
LTL-1006	5	034	Refrigerator, Freezer, and Oven Thermometer Calibration and Maintenance
LTL-1007	5	034	Use of Instrument Records and Logbooks
LTL-1008	7	034	QC Corrective Action
LTL-1009	3	034	Blind Spike Program
LTL-1011	7	034	Procedures for the Determination and Reporting of Detection Limits, Reporting Limits, Precision and Accuracy Studies, and Control Limits
LTL-1013	6	034	Preparation, Storage, Shelf Life and Traceability Documentation of Standards and Reference Materials
LTL-1017	3	034	Internal Audit Procedures
LTL-1018	3	034	Overview of Review and Approval Practices for Validatable Data Packages
LTL-1019	4	034	Controlling, Maintaining, and Monitoring Laboratory Logbooks
LTL-1020	2	034	Integration of IC, GC, HPLC and GCMS Peaks
LTL-2001	8	034	Waste Segregation and Disposal
LTL-4002	8	034	Electronic Sample Entry and Log-In
LTL-4201	0	034	Package Deliverables for all Reporting Levels
LTL-6006	1	034	Cation –Exchange Capacity of Soils (Sodium Acetate) by SW 846 Method 9081
LTL-6008	1	034	The Determination of Acid Volatile Sulfides and Simultaneously Extracted Metals in Sediment
LTL-7003	4	034	Inorganic Glass Cleaning Procedure
LTL-7015	3	034	Acid Digestion of Sediments, Sludges, and Soils Using SW846 Method 3050B
LTL-7202	7	034	Metals Analysis Using Inductively Coupled Plasma – Mass Spectrometry (ICP/MS) SW846 Method 6020
LTL-8000	4	034	Determination of Retention Time Windows
LTL-8330	16	034	Determination of Nitroaromatics and Nitramines by SW-846 Method 8330A & 8332

APPENDIX C

XRF INSTRUCTION MANUAL

INSTRUCTION MANUAL

INNOV-X SYSTEMS X-RAY FLUORESCENCE SPECTROMETERS

**October 2002
Version 1.1**

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1.0 INSPECTING YOUR INNOV-X ANALYZER

Upon receipt:

1. Locate and remove the shipping papers and documentation from under the lid's foam padding.
2. Remove the INNOV-X Analyzer and all of the components from the protective carrying case and identify each on the enclosed shipping list.
3. Connect the battery charger to an 110V-240V AC power source. Place one Li-ion battery on the charger and charge it for at least 2 hours. Charge the second battery.
4. Charge the Compaq iPAQ using attached AC adaptor for at least ½ hour.
5. Read and review the "Quick Start" section of the User's Manual. Innov-X recommends that you read the entire manual.
6. Install the fully charged battery into the analyzer
7. Press the ON/OFF button on the base of the pistol grip on the analyzer and the power button on the iPAQ
8. Select the desired analytical mode (i.e., Analytical, Fast ID, Pass/Fail or Soil). The instrument will undergo an approximately 3 minute warm-up period.
9. Standardize the instrument with the 316 Stainless Steel mask. Standardize the instrument every 4 hours or as directed by the display.
10. If possible, analyze a sample of known composition, in order to verify the correct operation of the analyzer.
11. Analyze samples of unknown metal composition.

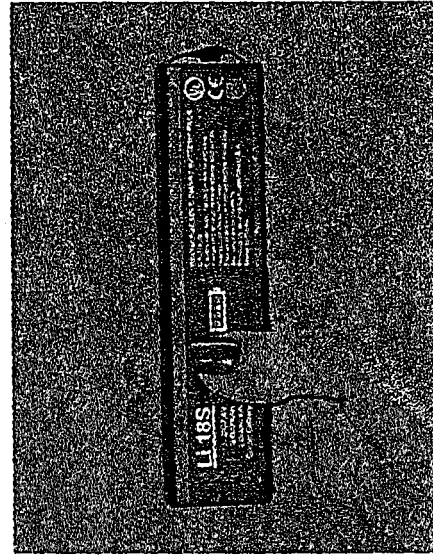
Note: If your analyzer has not been used for a week or more, you may have to charge the internal iPAQ Pocket PC battery as well. Please see Section 4.4 for instructions to recharge the iPAQ Pocket PC battery.

1.1 COMPONENTS INCLUDED WITH THE ANALYZER

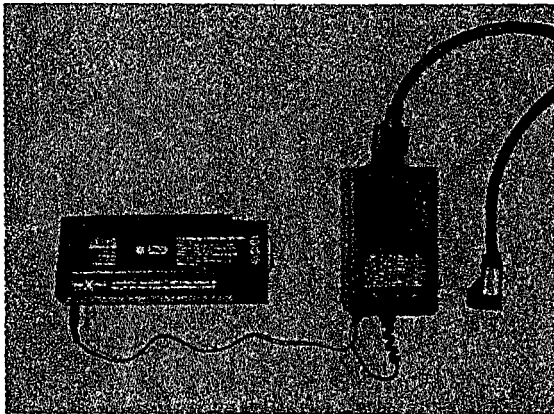
Shown here are the various items which are included in the Innov-X portable XRF analyzer. All items shown are included with every analyzer, with the exception of the test stand, which is an optional accessory.



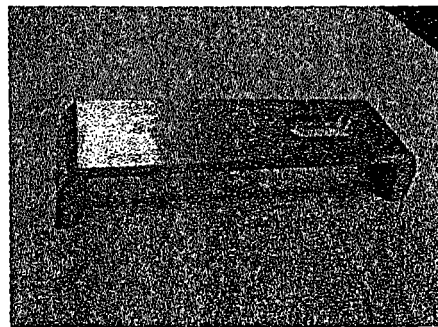
Analyzer, with iPAQ attached.



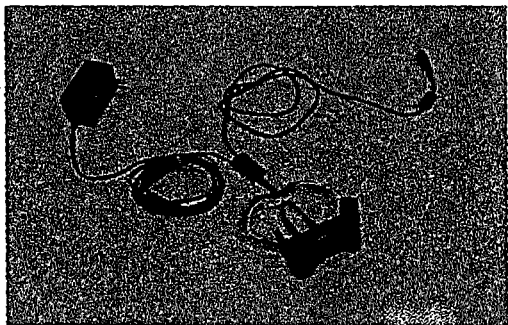
Two, Li-ion batteries (one shown).



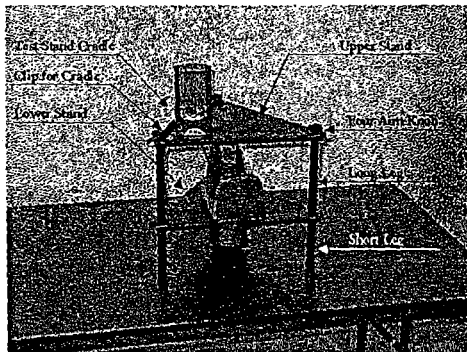
Battery charger and an AC adaptor. Battery shown mounted in charging system.



Standardization cap and mask for weld analysis.



IPAQ cradle and AC adapter. The cradle is used to connect the iPAQ to a PC for downloading data and reports.



Testing stand. This is the benchtop docking station for the analyzer. This accessory is standard with the XT-260 and the XT-440 (environmental analyzer).

1.2 QUICK START INSTRUCTIONS

The following section provides a quick overview to using your Innov-X portable XRF analyzer. This is intended to provide basic startup and operational instruction to perform basic alloy analysis. We highly recommend you read the sections on Radiation Safety (Chapter 2) and the detailed description on operation (Chapter 3). This following Quick Start pages are also available as a separate, bound, laminated publication as a stand-alone Quick Start Guide as well.

Power-On Procedure

1. Be sure Pocket PC is plugged attached to instrument body. This is the standard configuration for shipping from the factory.
2. Turn Main Power – ON; The switch is located at the base of the instrument.
3. Generally the Compaq IPAQ Pocket PC will also power on automatically unless. If the screen remains blank, press the small button on the top right of the Pocket PC to power ON.
4. Tap Microsoft icon at upper left corner of iPAQ.
5. Tap Innov-X logo to start software. You may see a message stating “Connecting to Host”. Ignore this – it is an internal feature of Microsoft Windows operating system.
6. Read User Authorization Dialogue Box & Choose “START”
7. Select desired analysis by tapping name on menu or by choosing Mode (bottom of screen) and then choose the desired mode from the drop-down menu.

e.g. Alloy Analysis

Fast ID
Pass/Fail
Analytical

Soil Mode

Note: Modes that are available on the analyzer are in blue, modes that are not available are in light gray.

8. The system will proceed with an automatic warm-up process requiring approximately 2 minutes. System will display bar graph indicating warm-up progress, with message stating “Initializing Hardware...”
9. Release manual trigger lock
10. Standardize on solid side of welding mask.
11. Remove mask, test alloy standard to check instrument performance
12. Place INNOV-X instrument on the test item, squeeze the trigger to begin test

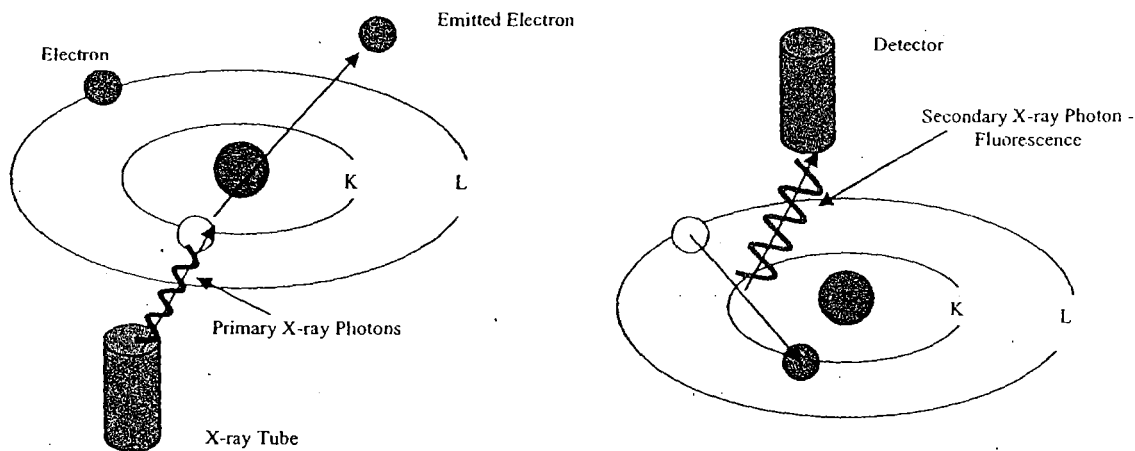
1.3 INTRODUCTION TO XRF: X-RAY FLUORESCENCE SPECTROMETRY OVERVIEW

Basic Theory

Although most commonly known for diagnostic use in the medical field, the use of x-rays forms the basis of many powerful analytical measurement techniques, including X-ray Fluorescence (XRF) Spectrometry.

XRF Spectrometry is used to identify elements in a substance and quantify the amount of those elements present. An element is identified by its characteristic X-ray emission wavelength (λ) or energy (E). The amount of an element present is quantified by measuring the intensity of its characteristic line. XRF Spectrometry ultimately determines the elemental composition of a material.

All atoms have a fixed number of electrons (negatively charged particles) arranged in orbitals around the nucleus. The number of electrons in a given atom is equal to the number of protons (positively charged particles) in the nucleus; and, the number of protons is indicated by the Atomic Number in the Periodic Table of Elements. Each Atomic Number is assigned an elemental name, such as Iron (Fe), with Atomic Number 26. Energy Dispersive (ED) XRF and Wavelength Dispersive (WD) XRF Spectrometry typically utilize activity in the first three electron orbitals, the K, L, and M lines, where K is closest to the nucleus. Each electron orbital corresponds to a specific and different energy level for a given element.



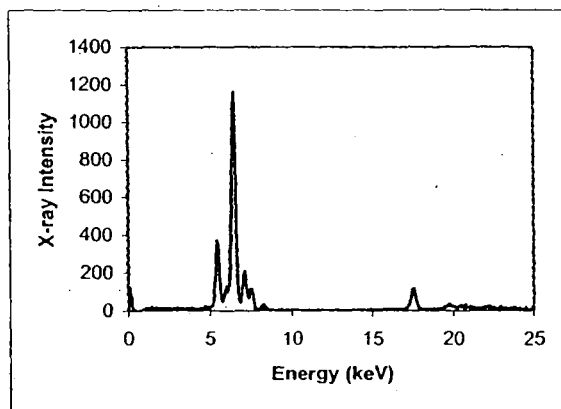
In XRF Spectrometry, high-energy primary X-ray photons are emitted from a source (X-ray tube) and strike the sample. The primary photons from the X-ray tube have enough energy to knock electrons out of the innermost, K or L, orbitals. When this occurs, the atoms become ions, which are unstable. Electrons seek stability; therefore, an electron from an outer orbital, L or M, will move into the newly vacant space at the inner orbital. As the electron from the outer orbital moves into the inner orbital space, it emits an energy known as a secondary X-ray photon. This phenomenon is called fluorescence. The secondary X-ray produced is characteristic of a specific element. The energy (E) of the emitted fluorescent X-ray photon is determined by the difference in energies between the initial and final orbitals of the individual transitions.

This is described by the formula

$$E=hc/\lambda$$

where h is Planck's constant; c is the velocity of light; and λ is the characteristic wavelength of the photon.

Wavelengths are inversely proportional to the energies; they are characteristic for each element. For example the $K\alpha$ energy for Iron (Fe) is about 6.4keV. The number of element-specific characteristic X-rays produced in a sample over a given period of time, or the intensity, can be measured to determine the quantity of a given element in a sample. Typical spectra for EDXRF Spectrometry appear as a plot of Energy (E) versus the Intensity (I).



History

Wilhelm Roentgen discovered X-rays in 1895. Methods for identifying and quantifying elements using XRF were first published by Henry Moseley in 1913. Much research and development of XRF continued after Moseley's pioneering work, especially during WWII when rapid developments in the aircraft, automotive, steel and other metals industries heightened the need to identify alloys quickly and reliably. However, the first commercial XRF Spectrometers weren't available until the early 1950's. Those systems were based on WDXRF technology and measured the characteristic wavelength of an element, one element at a time. Although the use of these systems was critical for elemental analyses, they were large, expensive, and required highly skilled operators to use and maintain them.

In the late 1960's, EDXRF technology, which measures the characteristic energy of an element, began to rival the use of WDXRF due to the development of Si (Li) solid state detectors, which offered better energy resolution of the signal. EDXRF systems offered the potential of collecting and displaying information on all of the elements in a sample at the same time, as opposed to one at a time with typical WDXRF systems. Many of the early EDXRF systems used radioisotopes for excitation instead of X-ray tubes, which could require changing sources to determine all the elements of interest. Some of those early EDXRF systems did not easily resolve multiple elements in a single analytical run.

As can be imagined, the equipment and applications of XRF Spectrometers have developed tremendously since the 1960's. Advancements in technology, electronics, computers, software and the use and modification of them for XRF Spectrometers by instrument manufacturers, research scientists & engineers, and industrial users alike have led to the current state of the art in XRF Spectrometers. Now a mature technology, XRF Spectrometry is routinely used for R&D, QC and analytical services in support of production.

Elemental Analysis

XRF Spectrometry is the choice of many analysts for elemental analysis when compared to the other techniques available. Wet chemistry instrument techniques for elemental analysis require destructive and time-consuming specimen preparation, often using concentrated acids or other hazardous materials. Not only is the sample destroyed, waste streams are generated during the analytical process that need to be disposed of, many of which are hazardous. These wet chemistry elemental analysis techniques often take

twenty minutes to several hours for specimen preparation and analysis time. All of these factors lead to a relatively high cost per sample. However, if PPB and lower elemental concentrations are the primary measurement need, wet chemistry instrument elemental analysis techniques are necessary.

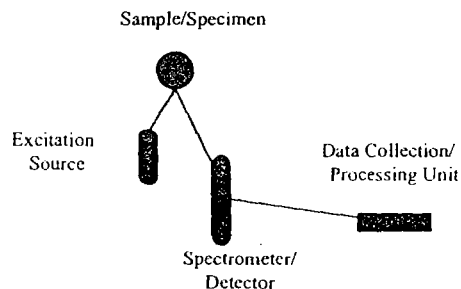
XRF Spectrometry easily and quickly identifies and quantifies elements over a wide dynamic concentration range, from PPM levels up to virtually 100% by weight. XRF Spectrometry does not destroy the sample and requires little, if any, specimen preparation. It has a very fast overall sample turnaround time. These factors lead to a significant reduction in the per sample analytical cost when compared to other elemental analysis techniques.

All elemental analysis techniques experience interferences, both chemical and physical in nature, and must be corrected or compensated for in order to achieve adequate analytical results. Most wet chemistry instrument techniques for elemental analysis suffer from interferences that are corrected for by both extensive and complex specimen preparation techniques, instrumentation advancements, and by mathematical corrections in the system's software. In XRF Spectrometry, the primary interference is from other specific elements in a substance that can influence (matrix effects) the analysis of the element(s) of interest. However, these interferences are well known and documented; and, instrumentation advancements and mathematical corrections in the system's software easily and quickly correct for them. In certain cases, the geometry of the sample can effect XRF analysis, but this is easily compensated for by grinding or polishing the sample, or by pressing a pellet or making glass beads.

Quantitative analysis for XRF Spectrometry is typically performed using Empirical Methods (calibration curves using standards similar in property to the unknown) or Fundamental Parameters (FP). FP is frequently preferred because it allows elemental analysis to be performed with no standards or calibration curves. This enables the analyst to use the system immediately, without having to spend additional time setting up individual calibration curves for the various elements and materials of interest. The capabilities of modern computers allow the use of this no-standard mathematical analysis, FP, accompanied by stored libraries of known materials, to determine not only the elemental composition of an unknown material quickly and easily, but even to identify the unknown material itself.

EDXRF Spectrometers

EDXRF Spectrometer systems mechanically very simple; essentially there are no moving parts. An EDXRF system typically has three major components: an excitation source, a spectrometer/detector, and a data collection/processing unit. The ease of use, rapid analysis time, lower initial purchase price and substantially lower long-term maintenance costs of EDXRF Spectrometers have led to having more systems in use today worldwide than WDXRF Spectrometer systems.



EDXRF has been found most useful for scrap alloy sorting, forensic science, environmental analysis, archaeometry and a myriad of other elemental field-oriented analyses.

Handheld EDXRF Spectrometers for Field Analyses

It is clear that a future trend for elemental analysis is in rapid site investigation using techniques that are fast, inexpensive, reliable, and long-term cost effective. There is a need for immediate decisions to be

made during the delivery of materials, industrial processing, and in the field for positive materials identification or environmental site assessment and remediation. It is also clear that EDXRF Spectrometry is the most suitable elemental analysis technique available for field analysis due to its simplicity, speed, precision, accuracy, reliability, and overall cost effectiveness.

Recent technological developments in cell phones, pocket PC's and other portable consumer electronics have led to the advancement of many high-performance, miniature components. X-ray equipment manufacturers began to take advantage of these developments in the late 1990's and developed Handheld EDXRF systems. An obvious advantage of Handheld EDXRF systems is that the analyzer is taken to the sample as opposed to bringing the sample to the analyzer and configuring it to fit in an analysis chamber. In addition to the per sample analytical cost savings, a key factor in using non-destructive EDXRF analysis, especially in the field, is the overall project cost savings due to improved and more timely decision making. The use of EDXRF for immediate positive materials identification or to guide an environmental site characterization will generally reduce the overall time required in the field due to the quick turnaround for the sample analysis; this invariably reduces the overall costs of analytical field work.

Of course, Handheld EDXRF technology has continued to evolve in concert with portable consumer electronic developments. Just like the early Benchtop EDXRF systems, early Handheld EDXRF systems used radioisotopes for excitation. There are several practical problems with the use of radioactive isotopes for handheld systems. The source decays and loses its testing speed over time. In addition to the loss in analytical capabilities, the sources have to be replaced incurring a cost. The use of radioactive isotopes also requires licensing (state-to-state in the US) and a radioactive materials control program; they are difficult to ship and transport, as they require hazardous materials declarations and/or permits. Consequently, the newest and most exciting development in Handheld EDXRF technology is the use of battery operated, miniature X-ray tubes, which was pioneered by the staff at Innov-X Systems.

Innov-X Systems Handheld EDXRF Spectrometers

Innov-X Systems specializes in Handheld EDXRF technology with the most advanced miniature components available for X-ray Tube sources, detectors, and PC 's. Innov-X Systems Handheld EDXRF Spectrometers are ideally suited for field analysis of alloys, lead-based paint, environmental soils, filters, dust wipes, forensics, archaeometry, and a variety of other elemental analyses in the field or around the plant. Innov-X Systems EDXRF Spectrometers are affordable, easy to use, reliable, and overall cost effective. The Innov-X Systems Handheld EDXRF units incorporate state-of the art components including a battery operated miniature X-ray tube, a high-resolution silicon pin detector, high speed data acquisition circuitry, and a Compaq IPAQ Pocket PC® handheld computer for calculations, results and operator interface.

Innov-X Systems EDXRF Spectrometers offer the following invaluable features:

- Portable
- Battery operated - rechargeable
- X-ray Tube-based (Ag anode, 10-35kV, 10-100uA)
- Si PiN diode detector (<250eV FWHM @5.90keV)
- Integrated pocket PC
- Pistol-shaped design for difficult testing locations and welds
- Auto-compensation for irregular or small samples
- Fundamental Parameters for no-standard analyses
- Stored Grade Libraries for rapid Grade ID's
- Stored Fingerprint Libraries for rapid material ID's
- Docking station available for use as standard benchtop unit
- Sample results within a few minutes after a few seconds of irradiation time

For more information on how to utilize your Innov-X Systems Handheld EDXRF Spectrometer optimally, please review this Instruction Manual or contact us directly.

3.0 Radiation Safety

3.0 IMPORTANT SAFETY INFORMATION

THE XRF SHOULD NOT BE POINTED AT ANYONE OR ANY BODY PART, ENERGIZED OR DE-ENERGIZED! The safe and proper operation of the INNOV-X XRF instruments is the highest priority. These instruments produce ionizing radiation and should **ONLY** be operated by individuals, who have been trained by INNOV-X Systems, Inc. and documented by a manufacturer's training certificate. INNOV-X recommends that operators and companies implement a written Radiation Safety Program, with safety components specific to the site and application of use of the instrument. The Radiation Safety Program should be reviewed annually and revised appropriately by a competent individual.

3.1 GENERAL SAFETY PRECAUTIONS

Retain and follow all product safety and operating instructions. Observe all warnings on the product and in the operating instructions. To reduce the risk of bodily injury, electric shock, fire and damage to the equipment, observe the following precautions:

Heed service markings. Except as explained in this documentation, do not service any INNOV-X product yourself. Opening or removing covers may expose you to electric shock. Service needed on components inside these compartments should be done only by INNOV-X Systems, INC.

Damage requiring service:

- The power cord, plug or battery contacts for the battery charger are damaged.
- Liquid has been spilled or an object has fallen onto the instrument.
- The instrument has been exposed to rain or water.
- The instrument has been dropped or damaged.
- There are noticeable signs of overheating.
- The instrument does not operate normally when you follow operating instructions.

Safety Precautions:

Use the correct external power source: Ensure that the voltage is appropriate (100V-240 V/ 50-60 Hz) for charging the battery packs. Do not overload an electrical outlet, power strip, or convenience receptacle. The overall load should not exceed 80% of the branch circuit rating.

Use cables and power cords properly:

Plug the battery charger into a grounded electrical outlet that is easily accessible at all times. Do not pull on cords and cables. When unplugging the cord from the electrical outlet, grasp and pull the cord by the plug.

Handle battery packs properly; do not: disassemble, crush, puncture, short external contacts, dispose of in fire or water, or expose a battery pack to temperatures higher than 60 °C (140 °F). Do not attempt to open or service a battery pack.

WARNING: Danger of explosion if battery is incorrectly substituted. Replace only with INNOV-X specified batteries. Dispose of used batteries in according to the information in the instruction manual supplied with your instrument.

3.2 INNOV-X SYSTEMS – RECOMMENDED RADIATION SAFETY TRAINING COMPONENTS

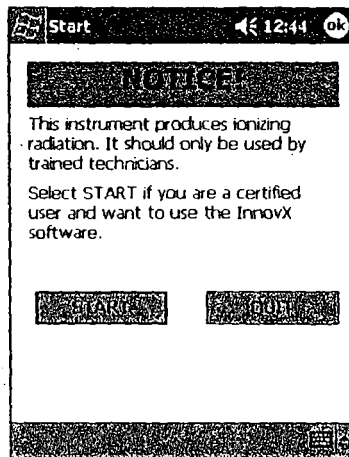
Individual Companies and States have specific regulations and guidelines for the use of X-ray tube generated ionizing radiation. The purpose of the recommendations below is to provide generic guidance for an ALARA - best practice - approach to radiation safety. These recommendations do not replace the requirement to understand and comply with the specific policies of any state or organization.

1. **Proper Usage.** Never point the instrument at another person. Never point the instrument into the air and perform a test. Never hold a sample in your hand and test that part of the sample.
2. **Establish Controlled Areas.** The location of storage and use should be of restricted access to limit potential exposure to ionizing radiation. In use, the target should not be hand held and the area at least three paces beyond the target should be unoccupied.
3. **Specific Controls.** The instrument should be stored, in a locked case, or locked cabinets when not in use. When in use, it must remain in the direct control of a factory trained, certified operator.
4. **Time - Distance - Shielding Policies.** Operators should minimize the time around the energized instrument, maximize the distance from the instrument window, and shoot into high density materials whenever possible. Under no circumstances should the operator point the instrument at themselves or others.
5. **Prevent Exposure to Ionizing Radiation.** - All reasonable measures, including labeling, operator training and certification, and the concepts of time, distance, & shielding, should be implemented to limit radiation exposure to *as low as reasonably achievable* (ALARA).
6. **Personal Monitoring.** Radiation control regulations may require implementation of a radiation monitoring program, where each instrument operator wears a film badge or TLD detector for an initial period of 1 year to establish a baseline exposure record. Continuing radiation monitoring after this period is recommended, but may be discontinued if accepted by radiation control regulators. Please refer to Sect. 3.10 for a list of providers of film badges.

3.3 PERFORMING A TEST FOLLOWING APPROPRIATE RADIATION SAFETY PROCEDURES

Starting up the Analyzer:

When an operator opens the Innov-X software on the iPAQ, he or she will be presented with the display shown below. Provided an operator has received training by an authorized person at the company, or from an authorized Innov-X trainer, they should touch the START icon to begin using the analyzer. From this point the operator is presented with the main menu of the analyzer to choose an operating mode and begin testing (described in Chapter 3). The remainder of this section is dedicated to operational and safety aspects that pertain to safe use and storage of the analyzer.



Starting a test using the trigger.

When the trigger is depressed, the analyzer supplies power to the x-ray tube and opens the shutter to emit x-rays. The analyzer is provided with a trigger lock to prevent the user from accidentally beginning a test. Slide the lock from left to right to lock the trigger.

Recommended Operation: When the system is not in use, slide the trigger lock to the right to “lock” the trigger. This will prevent a person from starting a test accidentally when they grab the analyzer. The location of the trigger lock is shown to the right.

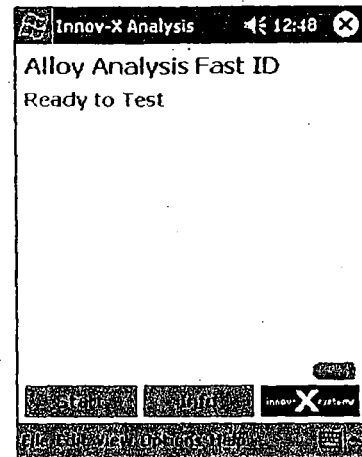
The trigger functions as both an “ON” and “OFF” switch. Touch the trigger once to begin a test. Do NOT hold the trigger as this will immediately stop the test. To end a test manually, press the trigger again.



Figure 3.1 ON/OFF Switch

Starting a Test Using the “Start” Icon on the iPAQ Screen

An operator may also begin a test by pressing the “Start” icon on the touch screen, as shown at the right. The “Start” icon, rather than the trigger, is generally used when the analyzer is docked into the testing stand. As an additional safety feature, the “Start” icon also provides for “two handed” operation of the analyzer. To require two-handed operation, set the trigger lock by sliding the switch left to right. This will require the user to hold the analyzer with one hand and start a test by using the other hand to press the ON switch.



3.4 CORRECT AND INCORRECT INSTRUMENT USAGE:

The Innov-X alloy analyzer can be used in several different testing configurations. Safety guidelines are described for each configuration.

Configuration 1: Handheld Alloy Analyzer:

In this configuration the analyzer is held in the hand, placed on various types of samples and a test is performed. Samples include pipes, valves, large pieces of scrap metal, basically any sample large enough to be tested in place, rather than held in the operator's hand. Point the instrument at a metal sample such that no part of your body including hands and/or fingers is near the aperture of the analyzer where x-rays are emitted.

Configuration 2: Testing Stand for small samples.

Innov-X strongly recommends that testing small pieces or small samples (rod, fasteners, turnings, XRF sample cups, bagged samples, etc.) be analyzed using the Innov-X Testing Stand. This allows the sample to be placed onto the analysis window of the analyzer without requiring the sample to be held by the operator. See figure below titled “Testing Stand Operation.”

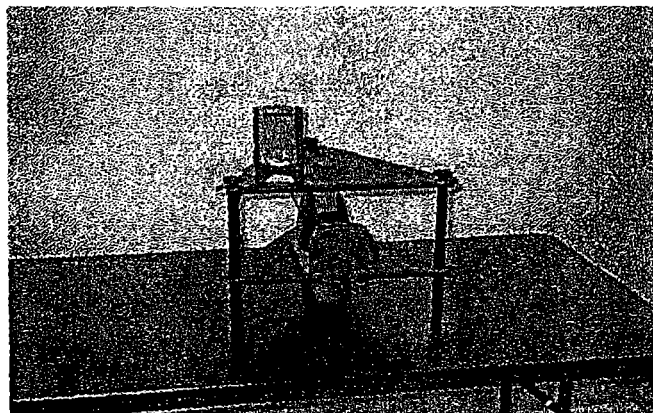


Figure 3.2 Testing Stand Operation. Please refer to Section 6.5 for instructions to assemble the test stand.

Warning: Innov-X strongly recommends that operators do NOT hold samples in their hand for testing. Never hold a small sample in your hand, and test that sample, such that your hand is exposed to the x-ray beam being emitted from the analyzer. This type of testing produces a small but non-negligible radiation dose to the operator's hand. Please see Section 3.5 Radiation Dose Examples for dose levels. See figure below titled "Incorrect Operation."

In the event that an operator insists on testing small parts by holding them in his/her hand, follow the precautionary procedures listed below. We again emphasize that this type of testing is recommended to be performed in a testing stand.

Proper Operation:

Hold the sample such that the operator's hand is not in front of the window of the analyzer, and is at least 3" away from the window. The figure below shows an acceptable means of testing a handheld sample. The operator should also be sure that other personnel are at least 6 feet away from the front end of the analyzer, to avoid any potential exposure. Exposure estimates for various scenarios are presented in the table below.



Figure 3.3. Innov-X always recommends using the testing stand for small parts. However, should an operator require holding a sample in his/her hand for analysis, the operators' hand should be at least 3" away from the instrument window and not in the direct path of the x-ray beam.

Improper Operation, DO NOT TEST SAMPLES LIKE THIS:

Never hold a sample in your hand such that any part of your body or appendages are exposed to the x-ray beam.

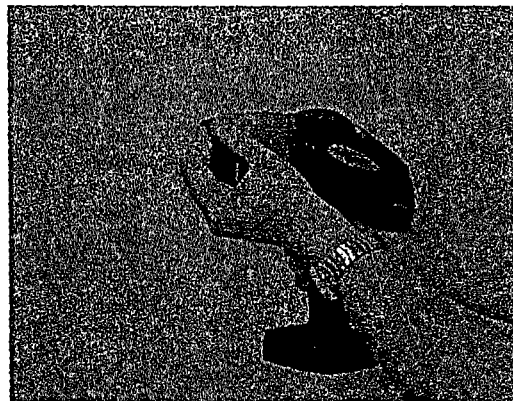


Figure 3.4. Example of INCORRECT usage of analyzer for testing small samples. Operators should never hold a sample in their hand to test.

3.5 RADIATION WARNING LIGHTS AND LABELING:

3.5.1 Probe Light and Probe Label:

The Innov-X analyzer is equipped with warning lights that alert the operator when the tube is receiving power, and when x-rays are being emitted from the analyzer. Please see Fig. 3.5 on the next page.

When the red light on the front nose of the analyzer is ON continuously (not blinking), this indicates the x-ray tube is receiving a low level of electrical power and the shutter is closed. The system is producing a low level of x-rays internally in this condition, but the shutter is providing adequate shielding to keep x-ray levels below levels of detection. The instrument is safe to be carried around or set down in this configuration.

When the red light is blinking, this indicates the tube is powered, the shutter is open and the analyzer is emitting x-ray radiation out of the analysis window. The analyzer should only be pointed at a sample, or be in the testing stand with a sample resting on the window, in this configuration.

3.5.2 Testing Light on Back of Analyzer:

The light on the back of the analyzer, shown in Fig. 3.6, is lit while a test is active. During a test – x-ray tube is energized and shutter is open – an LED on the back of the analyzer is lit. This LED remains lit until the test has ended. This light is for testing conditions (i.e. overhead) where the operator cannot see the Probe Light or the iPAQ display. The light turns off when the test is complete. When the light is off, the tube is de-energized and the shutter is closed.

3.5.3 Label Behind iPAQ:

The analyzer also has a label just below the iPAQ indicating, as shown in Figure 3.7:

CAUTION: Radiation. This Equipment Produces Radiation When Energized.

This label is required by most regulatory agencies. The term “When Energized” refers to the condition where the tube is fully energized and the shutter is open. This condition is also indicated by the red blinking light on the probe.

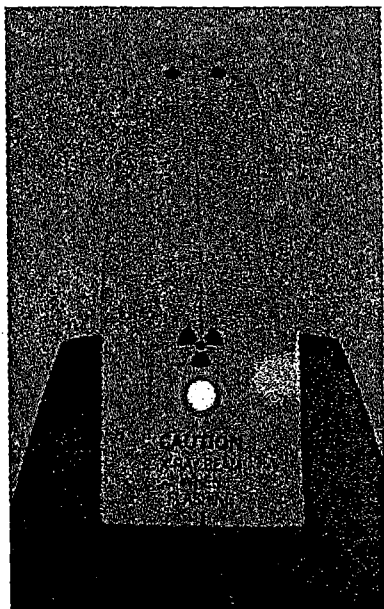


Figure 3.5. Probe light and labeling. When the light is on continuously, the x-ray tube is receiving minimal power and it is producing a minimum level of x-rays. The shutter is also closed so there is no radiation exposure to the operator or bystanders.

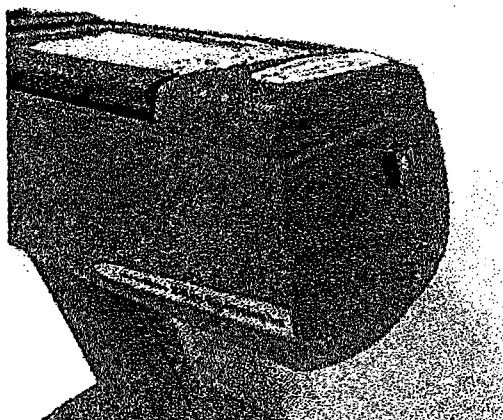


Figure 3.6. Back light on analyzer.



Figure 3.7. Label behind iPAQ.

3.6 RADIATION LEVELS FROM ANALYZER

Two pictures of the analyzer are shown below. In the first picture, all the relevant components referenced in this radiation safety section are displayed and labeled. The second picture shows a close-up of the front end of the window. The four sides A, B, C and D are indicated on this picture because they are referenced in terms of radiation levels output by the analyzer. The measured radiation levels for standard operating conditions are shown in the figures and tables below. Standard operating conditions are tube voltage operating at 35 kV, tube current of 30 uA, and 2 mm aluminum filtration.

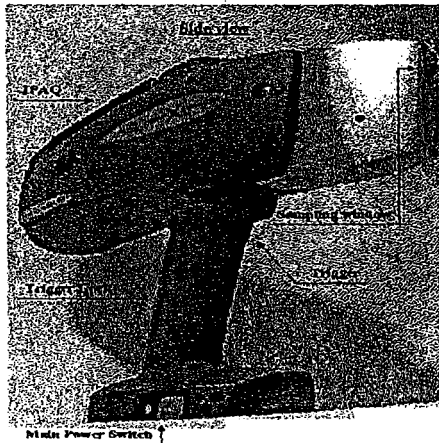


Figure 3.8 Innov-X Analyzer, Side View



Figure 3.9 Innov-X Analyzer, Front View

Sample at Window	Trigger	Location A (Top)	Location B (Right Side)	Location C (Bottom)	Location D (Left Side)
Blank (Air)	<0.05	2.0	2.1	0.15	1.5
Metal	<0.05	0.6	1.6	<0.05	1.6

Table 3.1. Dose rates at various locations with a metal sample covering the window and with no sample present. For “no sample” the analyzer is shooting the x-ray beam into air.

As shown in the Table 3-1, the dose to the operator’s hand is negligible. The radiation levels at the surface of the instrument snout are a maximum of 2.1 mrem/hour. Provided the operator follows standard operating procedures, there is no reason for any body part to be in the locations denoted A, B, C and D.

Table 3-2 shows the radiation levels directly in the x-ray beam that is emitted from the analyzer. Because the beam is collimated, it does not decline following the usual “r-squared” law. There is no reason for an operator to ever be exposed to this x-ray beam provided he/she is following standard operating procedures. The table entry “Trigger” shows the dose to the operators hand if a test is begun by pointing the analyzer into the air. There is no appreciable dose to the operator in this scenario. Table 2 also shows the dose rate received for a person being in the direct x-ray beam. Again, there is simply no operating procedure that would require an operator or a bystander to receive this dose rate.

Sample at Window	Trigger	At Window	12 inches	24 inches	36 inches	48 inches
Blank (Air)	<0.05	200	50	20	12	6

Table 3.2. Dose rates (units of mrem/hr) in the direct x-ray beam being emitted from the analyzer

3.7 RADIATION DOSES FOR SEVERAL SCENARIOS

In this section we provide data, concrete examples of use and misuse of the analyzer and common questions and answers we encounter when training personnel on the safe use of the Innov-X analyzer. The goals are to a) explain scenarios of safe versus improper usage of the analyzer and b) show that accidental exposures even in extreme situations are very low relative to the allowable dose to workers.

The table below presents radiation doses for normal operating conditions and also for examples of misuse of the analyzer and even extreme misuse. The purpose of these data is to demonstrate that even under conditions of extreme misuse; radiation dose to the operator is quite low relative to maximum allowable levels. Innov-X provides installation training that includes detailed radiation safety training and documentation designed to prevent misuse of the analyzer.

Example of Instrument Usage	Radiation Exposure and Comments
<p>Normal Operation - Dose to Hand: User analyzes samples according to standard operating procedures described in this manual. Assumption: Operator using system with x-ray tube ON for 8 hours/day, 5 days/week, 50 weeks/year. (Practically constant usage).</p>	<p>Maximum exposure is to operator's hand, at the trigger. Exposure is < 0.05 mrem/hr. Annual exposure to hand is then < 100 mrem.</p> <p>Maximum exposure under OSHA regulations is 50,000 mrem annually. Thus continuous operation provides a dose that is at least 500 times lower than maximum allowed by OSHA.</p>
<p>Normal Operation - Dose to Torso: Analyzer is used under the same operating conditions described above.</p>	<p>Exposure to Torso is so low it cannot be measured. It is estimated at < 0.01 mrem/hr. Annual exposure using highly conservative operating conditions above is < 20 mrem. Maximum allowed is 5,000 mrem.</p>
<p>Misuse Example 1: Operator holds samples in front of window with fingers. Fingers not directly in the beam but are placed around the sides of the instrument "nose."</p>	<p>Dose to fingers and/or hand in this example is a maximum of 2.1 mrem/hour. Assume operator performs 100 tests/day in this example, with each test being 30 seconds each, and operator does this 250 days/year. Dose is about 440 mrem/year. Maximum allowed dose is 50,000 mrem/year.</p>
<p>Misuse Example 2: Operator places analyzer against body and pulls the trigger to start a test. Analyzer tests to preset testing time (usually 5-10 seconds) unless operator pulls trigger again to stop test. This applies to analyzer being in contact with operator or with bystander.</p>	<p>Dose at exit of sampling window is approximately 200 mrem/hr. Dose for a 10 second exposure with analyzer in contact with Torso: 0.55 mrem. Thus operator would have to repeat this example of gross misuse 9,000 times in a year to achieve the maximum allowable exposure of 5,000 mrem.</p>
<p>Misuse Example 3: Operator holds small parts in fingers for analysis and exposes fingers and/or hand to x-ray beam at the sampling window.</p>	<p>As in Example 2, dose to fingers is approximately 200 mrem/hr. For a 10 second exposure, the total dose to the fingers or hand is 0.55 mrem. Thus operator would have to misuse analyzer in this fashion 90,000 times to achieve maximum allowed annual exposure.</p>

Misuse Example 4:

In the process of picking up or handling analyzer when it is powered on, operator forgets to lock the trigger and accidentally presses the trigger thus initiating a test. What is exposure to bystander?

Assume the analyzer is powered on for 30 seconds before operator realizes this problem, and assume bystander is actually standing in the x-ray beam at a distance of one foot.

Dose to bystander at 1 foot is 50 mrem/hr. For a 30 second exposure dose is 0.4 mrem. Maximum allowable level is 5,000 mrem assuming bystander's torso is exposed. Thus, this misuse would have to occur 12,500 times in a year to the same bystander before that bystander achieved his maximum allowed dose.

Misuse Example 5:

Operator manages to turn on analyzer and initiate a test, set it aside so it is emitting x-rays, and sets testing time to several hours (as opposed to the 5-30 seconds typical of various applications). What is maximum exposure situation?

Note: This example is almost impossible to conceive of in any real-world scenario. We include it as an example of how even the worse case scenario still does not provide significant exposure to an operator.

After 3 hours of continuous operation, analyzer's battery will be fully discharged. This will shut off analyzer entirely. The analyzer will not be able to produce x-rays until a new battery is provided and system is standardized. Maximum dose to operator is estimated at:

Operator 1 foot from analyzer: 150 mrem.

Operator at sampling window: 600 mrem.

Maximum allowable dose annually is 5,000 rem to torso, 50,000 to extremities.

Comparative: Radiation Doses from Typical Exposures to Ionizing Radiation

Common medical and/or dental x-rays:	20-30 mrem each.
Mammogram:	100-200 mrem
Flying in a commercial jet coast to coast (6 hrs.):	1-2 mrem.
Daily exposure from background radiation: * depends on geographic location	0.3 to 0.5 mrem/day

Table 3.3 Radiation Doses from Typical Exposures to Ionizing Radiation

From the above table, nearly all the cases of analyzer misuse produce radiation exposures similar to flying in a commercial aircraft. Moreover, the typical exposure from a case of significant instrument misuse produces roughly the same dosage as the daily dose of radiation from naturally occurring background radiation.

3.8 COMMON QUESTIONS AND ANSWERS REGARDING RADIATION SAFETY

Question: When I'm shooting a piece of pipe or valve on a rack or on a table top, is there any exposure to people standing in other locations, or standing several feet away from the analyzer?

Answer: Even a thin amount of metal sample (1-2 mm thickness) is enough to completely attenuate the x-ray beam emitted from the Innov-X analyzer. Shooting a piece of material that covers the sampling window on the analyzer will completely shield any bystanders from radiation exposure.

Question: If I forgot to switch the safety on the trigger to "ON", I pick up the analyzer and accidentally pull the trigger, is that dangerous to nearby personnel?

Answer: No, this example of misuse is not dangerous, but it may produce a non-negligible radiation exposure to nearby personnel. For an exposure to occur, the following things must happen. First, you must be holding the analyzer so that a bystander is actually standing in the x-ray beam being emitted. Just being near the analyzer is totally safe otherwise. Second, the bystander must be within 1-3 feet from the nose of the analyzer in addition to being in the beam path, to receive any appreciable dose. If all of these conditions are true, the dose received by a bystander is still extremely low. It ranges between 0.1 to 0.5 mrem depending on the exact location of the bystander. This dose is 10,000 to 50,000 times less than the allowed dose. Please see Misuse Example 4 in the table above.

Question: Do I need to create restricted areas where I am using the analyzer?

Answer: No, provided you are following normal operating procedures there is no reason to restrict access to an area where the analyzer is in use. The operator should take precautions to keep any personnel more than 3 feet away from the sampling window of the analyzer in the event of accidental misuse as detailed above. Should the operator also elect to test small components like weld rod as shown in Figure 3.3, the operator should also be sure that no personnel are standing within about 4-5 feet of the sampling window.

Question: How does the x-ray radiation from this analyzer compare to isotope-based systems?

Answer: The x-ray tube anode and power levels are designed to replicate the output from the typical isotopes used in portable XRF analyzers. Specifically, the output is designed to replicate the Cd-109 isotope and to some degree both the Fe-55 and Am-241 isotopes. By doing this, the analyzer can measure the same range of elements at the three isotopes listed. Moreover, the x-ray output from the analyzer, in units of mrem/hour, is very similar to the output of isotope based instruments. This is because the overall intensity and energy spectrum are designed to be similar to isotope systems.

Question: How does the x-ray tube in the Innov-X system compare to a radiography system used for taking images of metal parts.

Answer: The x-ray tube used in the Innov-X system produces between 1,000 and 10,000 times LESS radiation than most radiography systems. This is because a portable XRF is designed to perform surface analysis of alloys and other samples, whereas radiography systems are designed to shoot x-rays entirely through metal components in order to obtain an image on the other side of the object being bombarded with x-rays. For example, many tube-based radiography systems use a 300-400 kV tube and currents in the tens or hundreds of milliamps (mA). The Innov-X analyzer uses a tube operating at 34 kV and 20-30 micro-amps. The radiation levels produced are therefore thousands or tens of thousands times lower with the Innov-X system.

Question: Should we use dosimeter badges with the Innov-X analyzer.

Answer: Dosimeter badges are required by some states, and optional by other states. Innov-X recommends that operators wear badges, at least for the first year of operation, as a general precaution to flag any misuse of the analyzer. Dosimeter badges are available for the torso (generally worn on the belt loop or shirt pocket) and are available as “ring” badges. The best single badge to obtain is a ring badge that is worn on a finger, on the opposite hand used to hold the analyzer. This will record accidental exposure for the most likely case – an operator grabbing a small sample and holding it in one hand while analyzing it. Note: these badges generally have a threshold of 10 mrem, and are renewed monthly. So it will take several cases of misuse even to obtain a reading on a typical badge. When purchasing a badge, obtain the type used for x-ray and low energy gamma ray radiation.

3.9 SAFE GUARDS AND EMERGENCY RESPONSE

The main safeguards to use as an owner of an Innov-X portable XRF are really intended to restrict access to properly trained operators:

1. Keep the system in a controlled location, where only authorized users are likely to have access to the analyzer at any given time.
2. Make a simple sign that is kept with the analyzer indicating that an operator must have completed a training class provided by your company or must have attended an Innov-X training course in order to use the analyzer. Note that when the Innov-X system is turned on, the screen displays a message indicating that the system should only be used by authorized personnel.

Emergency Response:

Because the Innov-X system is a battery operated, x-ray tube based analyzer, the emergency response plan is very simple. If the operator believes the analyzer is locked up in an "OPEN" position, they should do two things:

1. Press the On/Off switch on the base to power the analyzer off. The green LED indicator will turn off, indicating system power is off. At this point it is not possible for the analyzer to be producing x-rays.
2. As an additional precaution, the operator may remove the battery trap door at the bottom of the analyzer (have the nose pointing away from personnel), and pull out the battery. Even if the operator has failed to properly power the system off in Step #1, removing the battery guarantees that no x-rays can be produced. There is no electrical power being provided to the x-ray tube.

Note: It would be highly unusual for an operator to somehow lock up the analyzer with the x-ray tube powered on. This would require the operator to crash the iPAQ during an analysis. If this happens the analyzer will shut off the x-ray tube 10 seconds after the last communication with the iPAQ. However, if at any time the operator believes the x-ray tube is on and no test is in progress, powering off the analyzer and restarting will automatically shut down the x-ray tube and close the shutter. It will no longer be possible to produce x-rays at this point.

3.10 DOSIMETER BADGES

Dosimeter badges are provided as a monthly service by several companies, listed in this section (see below). The badges are generally provided monthly, and the operator returns the previous month badges to the company for analysis. The operator receives a monthly report showing any personnel with readings higher than typical background radiation.

Dosimeter badges are required by some states, and optional by other states. Innov-X recommends that operators wear badges, at least for the first year of operation, as a general precaution to flag any misuse of the analyzer. Dosimeter badges are available for the torso (generally worn on the belt loop or shirt pocket) and are available as "ring" badges. The best single badge to obtain is a ring badge that is worn on a finger, on the opposite hand used to hold the analyzer. This will record accidental exposure for the most likely case – an operator grabbing a small sample and holding it in one hand while analyzing it. Note: these badges generally have a threshold of 10 mrem, and are renewed monthly. So it will take several cases of misuse even to obtain a reading on a typical badge. When purchasing a badge, obtain the type used for x-ray and low energy gamma ray radiation.

Dosimeter Companies:

Here are two companies that provide badges as a regular service. There are certainly many more.

Landauer Inc.
Glenwood, IL
708-755-7000

AEIL
Houston, TX
713-790-9719

3.11 TYPICAL REGISTRATION REQUIREMENTS

Innov-X maintains a database of the registration requirements for every state, including sample registration forms. Most states require some form of registration, and generally they require the registration to be received within 30 days of receipt of the instrument. Some states require no registration, while a few require notification in advance. Please contact Innov-X for specific questions regarding the state where the instrument will be used, or for copies of registration forms.

In general a company will have to provide the following information regarding the device:

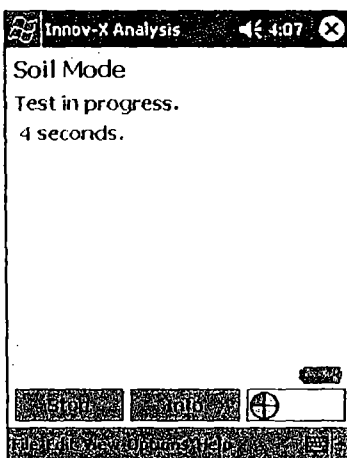
1. Purpose of device. Generally this is "Analytical" or "Industrial." Be sure to inform the state registration office that the device will NOT be used for radiography or for medical uses.
2. Radiation Safety Officer – Monitors training, safe use, and controls access to the instrument.
3. Authorized Users – Trained by Innov-X Factory Authorized Representatives in the safe and proper use of the XRF.
4. Operating parameters of the analyzer – 35 kV, 30 micro-amps.
5. Type of system, either fixed, mobile or portable. Generally the correct choice is "Portable."
6. User Training Specified – Indicate that only individuals receiving manufacturer training, documented by a manufacturer's training certificate will operate the instrument.
7. Personal Monitoring. This may be required by radiation control authorities. Many registration forms will ask that you indicate whether or not you intend to perform dosimeter monitoring.
8. Copy of Registration & Manual at the Job Site

If you have any questions regarding the type of registration form or filling out the form, please contact Innov-X Systems. Many states may confuse a portable XRF system that uses a tube with medical or industrial radiography systems. This is because of the relative newness of portable tube-based systems. In all likelihood, Innov-X personnel have experience providing the necessary documentation to the state in question, and can readily assist the customer in this process.

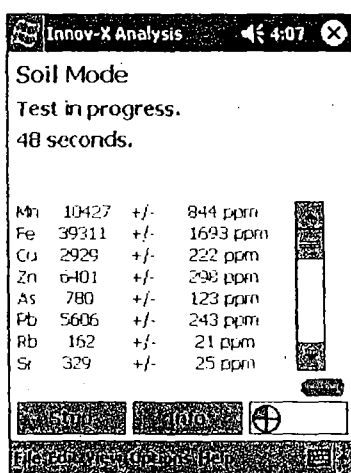
5.0S Soil Analysis

5.0 TESTING IN SOIL MODE

After the instrument has been standardized, testing can begin. Simply pull the trigger or press **Start** on the iPAQ screen to begin the test. The red warning light on the top of the instrument will blink, indicating X-rays are being emitted. The screen will display the words "Test in progress" and the time elapsed. The word "Testing" will blink on and off in the low right hand corner of the screen.

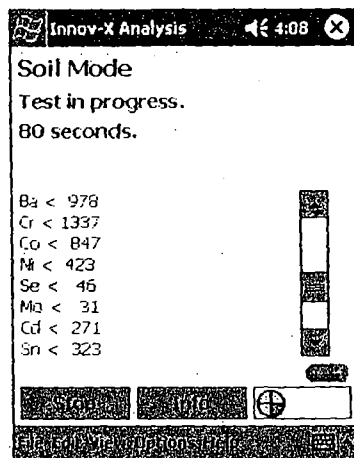


After a minimum time has elapsed, intermediate results will be displayed on the screen. This minimum time can be set by the user by selecting *Options* → *Setup Testing*. More details about this function can be found in the section **Soil Mode Options**. Each line of the results display shows the name of an element, its calculated concentration and the error on the measurement. This error is defined to be sigma, the error on the counting statistics of the measurement. The error will decrease with increased testing time.



Too many elements are measured in soil mode to display them at one time. As a result, it is possible to use the scroll bar located to the right of the chemistry display to view other elements. The complete display

shows detected elements first, listed in order of atomic number, from lightest to heaviest. Following the detected elements are the elements which are below the detection limit of the instrument. These elements are shown as less than a calculated LOD. This LOD is defined as three times the error on the counting statistics of the measurement.



When the measurement is complete, the results screen will open displaying the final results of the measurement.

5.1 SOIL RESULTS SCREEN

The standard Soil Mode results screen displays the concentration in ppm and error in measurement for detected elements, followed by the list of non-detected elements with the calculated limit of detection for each element for the test. If the display does not show soil chemistry results, change the display by selecting either *View* → *Results* or *View* → *Chemistry*.

Innov-X Results			
4:09			
Reading #35 07/08/2002			
Soil Mode			
Detected	ppm	+/-	
Mn	10382	502	
Fe	39180	1020	
Cu	2832	133	
Zn	6730	186	
As	777	71	
Pb	5492	144	
Rb	144	12	
Sr	328	15	
Not Detected ppm			
Ti	< 3509		
Ba	< 825		
Cr	< 1124		
Co	< 600		

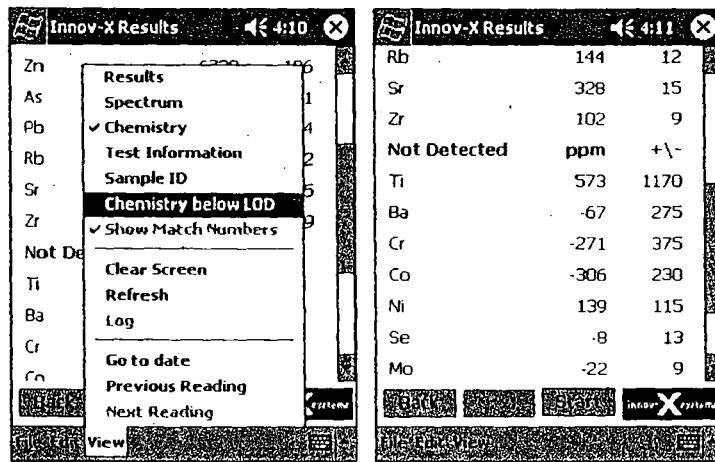
Innov-X Results			
4:09			
Reading #35 07/08/2002			
Soil Mode			
Detected	ppm	+/-	
Mn	10382	502	
Fe	39180	1020	
Cu	2832	133	
Zn	6730	186	
As	777	71	
Pb	5492	144	
Rb	144	12	
Sr	328	15	
Not Detected ppm			
Ti	< 3509		
Ba	< 825		
Cr	< 1124		
Co	< 600		

5.1.1 Results View Menu

The standard soil chemistry display can be modified by using the View Menu. As with all Innov-X software, it is possible to view spectra and Test Information. In addition, it is possible to view calculated chemistry below LOD.

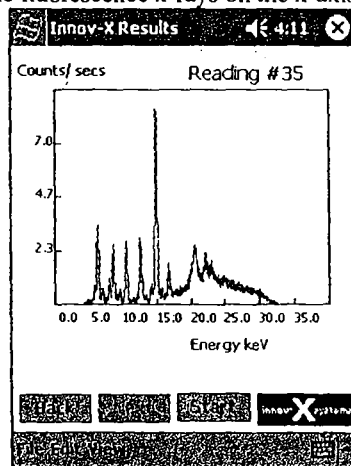
5.1.2 Chemistry Below LOD

A few select users have requested the ability to be able to see what the calculated chemistry would be for elements below detection limit. This can be done by selecting **View → Chemistry below LOD**. When this is done, the Elements below LOD are shown as a value in ppm, with an error. This is mainly for statistical purposes. These data should not be considered to be valid calculations. As a result, it is recommended that most users do not use this option.

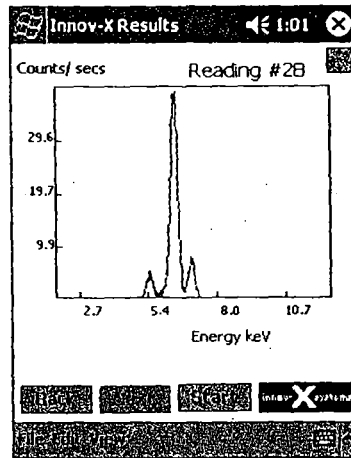
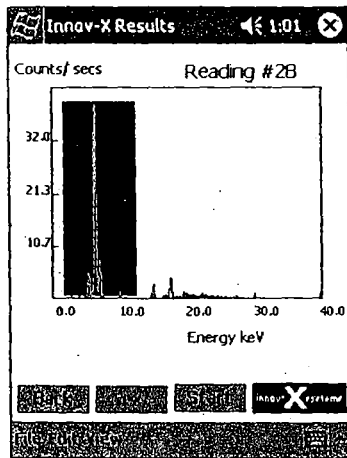


5.1.3 Spectrum Screen

This screen displays a plot of the x-ray fluorescence spectrum for an individual test, plotting the intensity on the y-axis versus the energy of the fluorescence x-rays on the x-axis.



Tapping on the spectra will show the energy scale and counts rate at the selected point. It is possible to zoom in on certain areas of the graph by selecting one corner and drawing out the out the region



Tapping the symbol in the upper right hand corner beneath the X will restore the graph to full scale.

5.1.4 Test Info Screen

The test information screen shows any information that was entered in TEST INFORMATION before starting a test.

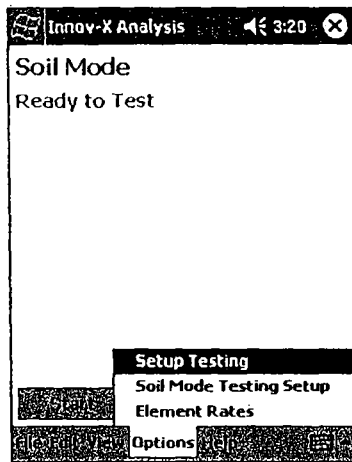
5.2 SOIL MODE OPTIONS

The length of tests in Soil Mode is user-settable. Users may select a minimum testing time, and as well as choose from a variety of test end conditions.

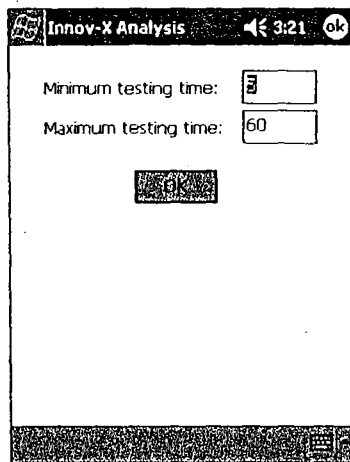
The options related to test time are contained in two menus: *Options* → *Setup Testing*, and *Options* → *Soil Mode Setup Testing*. *Setup Testing* contains minimum and maximum testing time information, while *Soil Mode Setup Testing* allows the user to select test end conditions.

5.2.1 Setup Testing Option—setting minimum and maximum testing times

To access the Setup Testing screen, select *Options* → *Setup Testing*



A screen appears prompting you to enter a Minimum and Maximum Testing time.



The minimum testing time is the required time that must elapse before results can be calculated. Live Update results will not be displayed on the screen until the minimum has elapsed, likewise a test must complete the minimum time before any test end condition can be used. If a test is stopped before the minimum testing time has elapsed, the test will be aborted, and no results will be calculated.

Maximum testing time is relevant only if "Maximum Testing Time" is selected from Soil Mode Setup Testing. This will automatically end the test at a preset testing time. Typically, the maximum testing time will be in excess of 30 seconds, and may be 1 or 2 minutes, depending on detection limits and desired precision.

It should be noted, that all testing times refer to "Real Time," the time the measurement takes when timed on a normal clock. There is some detector dead time associated with a measurement so the length of the test stored in the analyzer may be slightly shorter than the preset time.

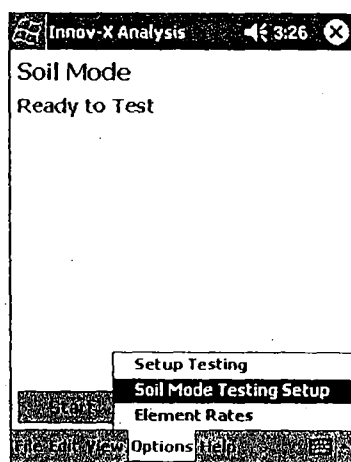
5.2.2 Soil Mode Testing Setup Option—Setting test end criteria

Four options exist for the test end criteria in soil mode. Depending on your application, you may choose to end the test manually, at a preset testing time, or when the uncertainty in the measurement is within a

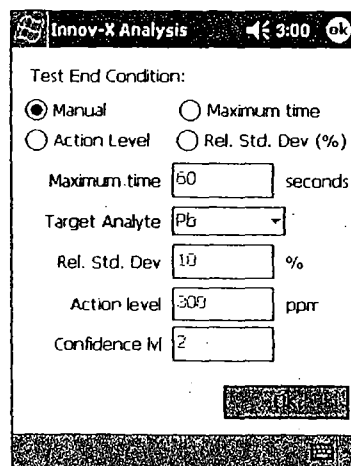
specified relative standard deviation of the reading. Additionally, you can set up an action level for a single element. As soon as the measuring statistics are good enough to ensure that the reading is above, below or at the action level, the test will end automatically. This allows for very rapid tests for elements that are well above or below an action level.

In all modes, pressing Stop, or pulling the trigger will end the test. If the minimum testing time has elapsed, results will be calculated. Otherwise the test will be aborted without calculating results.

Manual: This option allows you to look at the results which are being continually updated on the screen and determine when the results look satisfactory. The test will continue until the trigger is pulled, or Stop is tapped on the iPAQ screen. Results will be calculated if the testing time has exceeded the Minimum Test time which is set up in *Options* → *Setup Testing*. In order to preserve battery life, the software will stop if the testing time exceeds 300 seconds, since there is little to no advantage to continuing a test beyond 300 seconds.



To use Manual Test End Condition, simply choose *Options* → *Soil Mode Testing Setup* and select **Manual**. Press **ok** to return to the analysis screen.



Maximum Time: If Maximum Time is selected, the test will continue until the preset Time is reached. This is useful if you wish to do a set of measurements with the same testing time.

Innov-X Analysis 2:59 OK

Test End Condition:

☐ Manual ☒ Maximum time

☐ Action Level ☐ Rel. Std. Dev (%)

Maximum time 60 seconds

Target Analyte Pb

Rel. Std. Dev 10 %

Action level 300 ppm

Confidence Int 2

OK

To choose to end test based on a maximum time, select *Options* → *Soil Mode Testing Setup* and tap “Manual”. Enter the desired testing time in the appropriate test box. Tap ok to save your selections.

Action Level: System ends test when result for target analyte including chosen precision level is above or below pre-set action level.

Innov-X Analysis 3:02 OK

Test End Condition:

☐ Manual ☐ Maximum time

☒ Action Level ☐ Rel. Std. Dev (%)

Maximum time 60 seconds

Target Analyte Pb

Rel. Std. Dev 10 %

Action level 300 ppm

Confidence Int 2

OK

To choose to end a test based on an Action Level, select *Options* → *Soil Mode Testing Setup* and tap “Action Level.” Select a target analyte, specify an action level in ppm, and a confidence level. This confidence level refers to the number of sigma required for the precision. This should typically be set to 2. Tap ok to save your selections.

Relative Standard Deviation (RSD): When RSD is selected as a test end criterion; the system will end a test when the relative standard deviation on a target analyte reaches a pre-set level. This standard deviation is specified as a percentage of the reading. For example, if the measured value for an analyte was 1000 ppm, and the RSD was set to 10, the reading would stop when the error reached 100 ppm, or 10% of 1000.

Innov-X Analysis 3:02 ok

Test End Condition:

☐ Manual
 ☐ Maximum time
 ☒ Rel. Std. Dev (%)
 ☐ Action Level

Maximum time seconds

Target Analyte

Rel. Std. Dev %

Action level ppm

Confidence M

To choose to end a test based on a Relative Standard Deviation, select **Options** → **Soil Mode Testing Setup** and tap “Rel. Std. Dev (%)”. Select a target analyte and the desired Relative Standard Deviation. Tap ok to save your selections.

METHOD 6200

FIELD PORTABLE X-RAY FLUORESCENCE SPECTROMETRY FOR THE DETERMINATION OF ELEMENTAL CONCENTRATIONS IN SOIL AND SEDIMENT

1.0 SCOPE AND APPLICATION

1.1 This method is applicable to the in situ and intrusive analysis of the 26 analytes listed in Table 1 for soil and sediment samples. Some common elements are not listed in Table 1 because they are considered "light" elements that cannot be detected by field portable x-ray fluorescence (FPXRF). They are: lithium, beryllium, sodium, magnesium, aluminum, silicon, and phosphorus. Most of the analytes listed in Table 1 are of environmental concern, while a few others have interference effects or change the elemental composition of the matrix, affecting quantitation of the analytes of interest. Generally elements of atomic number 16 or greater can be detected and quantitated by FPXRF.

1.2 Detection limits depend on several factors, the analyte of interest, the type of detector used, the type of excitation source, the strength of the excitation source, count times used to irradiate the sample, physical matrix effects, chemical matrix effects, and interelement spectral interferences. General instrument detection limits for analytes of interest in environmental applications are shown in Table 1. These detection limits apply to a clean matrix of quartz sand (silicon dioxide) free of interelement spectral interferences using long (600-second) count times. These detection limits are given for guidance only and will vary depending on the sample matrix, which instrument is used, and operating conditions. A discussion of field performance-based detection limits is presented in Section 13.4 of this method. The clean matrix and field performance-based detection limits should be used for general planning purposes, and a third detection limit discussed, based on the standard deviation around single measurements, should be used in assessing data quality. This detection limit is discussed in Sections 9.7 and 11.3.

1.3 Use of this method is restricted to personnel either trained and knowledgeable in the operation of an XRF instrument or under the supervision of a trained and knowledgeable individual. This method is a screening method to be used with confirmatory analysis using EPA-approved methods. This method's main strength is as a rapid field screening procedure. The method detection limits (MDL) of FPXRF are above the toxicity characteristic regulatory level for most RCRA analytes. If the precision, accuracy, and detection limits of FPXRF meet the data quality objectives (DQOs) of your project, then XRF is a fast, powerful, cost effective technology for site characterization.

2.0 SUMMARY OF METHOD

2.1 The FPXRF technologies described in this method use sealed radioisotope sources to irradiate samples with x-rays. X-ray tubes are used to irradiate samples in the laboratory and are beginning to be incorporated into field portable instruments. When a sample is irradiated with x-rays, the source x-rays may undergo either scattering or absorption by sample atoms. This latter process is known as the photoelectric effect. When an atom absorbs the source x-rays, the incident radiation dislodges electrons from the innermost shells of the atom, creating vacancies. The electron vacancies are filled by electrons cascading in from outer electron shells. Electrons in outer shells have higher energy states than inner shell electrons, and the outer shell electrons give off energy as they cascade down into the inner shell vacancies. This rearrangement of electrons

results in emission of x-rays characteristic of the given atom. The emission of x-rays, in this manner, is termed x-ray fluorescence.

Three electron shells are generally involved in emission of x-rays during FPXRF analysis of environmental samples: the K, L, and M shells. A typical emission pattern, also called an emission spectrum, for a given metal has multiple intensity peaks generated from the emission of K, L, or M shell electrons. The most commonly measured x-ray emissions are from the K and L shells; only metals with an atomic number greater than 57 have measurable M shell emissions.

Each characteristic x-ray line is defined with the letter K, L, or M, which signifies which shell had the original vacancy and by a subscript alpha (α) or beta (β), which indicates the higher shell from which electrons fell to fill the vacancy and produce the x-ray. For example, a K_α line is produced by a vacancy in the K shell filled by an L shell electron, whereas a K_β line is produced by a vacancy in the K shell filled by an M shell electron. The K_α transition is on average 6 to 7 times more probable than the K_β transition; therefore, the K_α line is approximately 7 times more intense than the K_β line for a given element, making the K_α line the choice for quantitation purposes.

The K lines for a given element are the most energetic lines and are the preferred lines for analysis. For a given atom, the x-rays emitted from L transitions are always less energetic than those emitted from K transitions. Unlike the K lines, the main L emission lines (L_α and L_β) for an element are of nearly equal intensity. The choice of one or the other depends on what interfering element lines might be present. The L emission lines are useful for analyses involving elements of atomic number (Z) 58 (cerium) through 92 (uranium).

An x-ray source can excite characteristic x-rays from an element only if the source energy is greater than the absorption edge energy for the particular line group of the element, that is, the K absorption edge, L absorption edge, or M absorption edge energy. The absorption edge energy is somewhat greater than the corresponding line energy. Actually, the K absorption edge energy is approximately the sum of the K, L, and M line energies of the particular element, and the L absorption edge energy is approximately the sum of the L and M line energies. FPXRF is more sensitive to an element with an absorption edge energy close to but less than the excitation energy of the source. For example, when using a cadmium-109 source, which has an excitation energy of 22.1 kiloelectron volts (keV), FPXRF would exhibit better sensitivity for zirconium which has a K line energy of 15.7 keV than to chromium, which has a K line energy of 5.41 keV.

2.2 Under this method, inorganic analytes of interest are identified and quantitated using a field portable energy-dispersive x-ray fluorescence spectrometer. Radiation from one or more radioisotope sources or an electrically excited x-ray tube is used to generate characteristic x-ray emissions from elements in a sample. Up to three sources may be used to irradiate a sample. Each source emits a specific set of primary x-rays that excite a corresponding range of elements in a sample. When more than one source can excite the element of interest, the source is selected according to its excitation efficiency for the element of interest.

For measurement, the sample is positioned in front of the probe window. This can be done in two manners using FPXRF instruments: in situ or intrusive. If operated in the in situ mode, the probe window is placed in direct contact with the soil surface to be analyzed. When an FPXRF instrument is operated in the intrusive mode, a soil or sediment sample must be collected, prepared, and placed in a sample cup. The sample cup is then placed on top of the window inside a protective cover for analysis.

Sample analysis is then initiated by exposing the sample to primary radiation from the source. Fluorescent and backscattered x-rays from the sample enter through the detector window and are converted into electric pulses in the detector. The detector in FPXRF instruments is usually either a solid-state detector or a gas-filled proportional counter. Within the detector, energies of the characteristic x-rays are converted into a train of electric pulses, the amplitudes of which are linearly proportional to the energy of the x-rays. An electronic multichannel analyzer (MCA) measures the pulse amplitudes, which is the basis of qualitative x-ray analysis. The number of counts at a given energy per unit of time is representative of the element concentration in a sample and is the basis for quantitative analysis. Most FPXRF instruments are menu-driven from software built into the units or from personal computers (PC).

The measurement time of each source is user-selectable. Shorter source measurement times (30 seconds) are generally used for initial screening and hot spot delineation, and longer measurement times (up to 300 seconds) are typically used to meet higher precision and accuracy requirements.

FPXRF instruments can be calibrated using the following methods: internally using fundamental parameters determined by the manufacturer, empirically based on site-specific calibration standards (SSCS), or based on Compton peak ratios. The Compton peak is produced by backscattering of the source radiation. Some FPXRF instruments can be calibrated using multiple methods.

3.0 DEFINITIONS

3.1 FPXRF: Field portable x-ray fluorescence.

3.2 MCA: Multichannel analyzer for measuring pulse amplitude.

3.3 SSCS: Site specific calibration standard.

3.4 FP: Fundamental parameter.

3.5 ROI: Region of interest.

3.6 SRM: Standard reference material. A standard containing certified amounts of metals in soil or sediment.

3.7 eV: Electron Volt. A unit of energy equivalent to the amount of energy gained by an electron passing through a potential difference of one volt.

3.8 Refer to Chapter One and Chapter Three for additional definitions.

4.0 INTERFERENCES

4.1 The total method error for FPXRF analysis is defined as the square root of the sum of squares of both instrument precision and user- or application-related error. Generally, instrument precision is the least significant source of error in FPXRF analysis. User- or application-related error is generally more significant and varies with each site and method used. Some sources of interference can be minimized or controlled by the instrument operator, but others cannot. Common sources of user- or application-related error are discussed below.

4.2 Physical matrix effects result from variations in the physical character of the sample. These variations may include such parameters as particle size, uniformity, homogeneity, and surface condition. For example, if any analyte exists in the form of very fine particles in a coarser-grained matrix, the analyte's concentration measured by the FPXRF will vary depending on how fine particles are distributed within the coarser-grained matrix. If the fine particles "settle" to the bottom of the sample cup, the analyte concentration measurement will be higher than if the fine particles are not mixed in well and stay on top of the coarser-grained particles in the sample cup. One way to reduce such error is to grind and sieve all soil samples to a uniform particle size thus reducing sample-to-sample particle size variability. Homogeneity is always a concern when dealing with soil samples. Every effort should be made to thoroughly mix and homogenize soil samples before analysis. Field studies have shown heterogeneity of the sample generally has the largest impact on comparability with confirmatory samples.

4.3 Moisture content may affect the accuracy of analysis of soil and sediment sample analyses. When the moisture content is between 5 and 20 percent, the overall error from moisture may be minimal. However, moisture content may be a major source of error when analyzing samples of surface soil or sediment that are saturated with water. This error can be minimized by drying the samples in a convection or toaster oven. Microwave drying is not recommended because field studies have shown that microwave drying can increase variability between FPXRF data and confirmatory analysis and because metal fragments in the sample can cause arcing to occur in a microwave.

4.4 Inconsistent positioning of samples in front of the probe window is a potential source of error because the x-ray signal decreases as the distance from the radioactive source increases. This error is minimized by maintaining the same distance between the window and each sample. For the best results, the window of the probe should be in direct contact with the sample, which means that the sample should be flat and smooth to provide a good contact surface.

4.5 Chemical matrix effects result from differences in the concentrations of interfering elements. These effects occur as either spectral interferences (peak overlaps) or as x-ray absorption and enhancement phenomena. Both effects are common in soils contaminated with heavy metals. As examples of absorption and enhancement effects; iron (Fe) tends to absorb copper (Cu) x-rays, reducing the intensity of the Cu measured by the detector, while chromium (Cr) will be enhanced at the expense of Fe because the absorption edge of Cr is slightly lower in energy than the fluorescent peak of iron. The effects can be corrected mathematically through the use of fundamental parameter (FP) coefficients. The effects also can be compensated for using SSCS, which contain all the elements present on site that can interfere with one another.

4.6 When present in a sample, certain x-ray lines from different elements can be very close in energy and, therefore, can cause interference by producing a severely overlapped spectrum. The degree to which a detector can resolve the two different peaks depends on the energy resolution of the detector. If the energy difference between the two peaks in electron volts is less than the resolution of the detector in electron volts, then the detector will not be able to fully resolve the peaks.

The most common spectrum overlaps involve the K_{β} line of element Z-1 with the K_{α} line of element Z. This is called the K_{α}/K_{β} interference. Because the $K_{\alpha}:K_{\beta}$ intensity ratio for a given element usually is about 7:1, the interfering element, Z-1, must be present at large concentrations to cause a problem. Two examples of this type of spectral interference involve the presence of large concentrations of vanadium (V) when attempting to measure Cr or the presence of large concentrations of Fe when attempting to measure cobalt (Co). The V K_{α} and K_{β} energies are 4.95

and 5.43 keV, respectively, and the Cr K_{α} energy is 5.41 keV. The Fe K_{α} and K_{β} energies are 6.40 and 7.06 keV, respectively, and the Co K_{α} energy is 6.92 keV. The difference between the V K_{β} and Cr K_{α} energies is 20 eV, and the difference between the Fe K_{β} and the Co K_{α} energies is 140 eV. The resolution of the highest-resolution detectors in FPXRF instruments is 170 eV. Therefore, large amounts of V and Fe will interfere with quantitation of Cr or Co, respectively. The presence of Fe is a frequent problem because it is often found in soils at tens of thousands of parts per million (ppm).

4.7 Other interferences can arise from K/L, K/M, and L/M line overlaps, although these overlaps are less common. Examples of such overlap involve arsenic (As) K_{α} /lead (Pb) L_{α} and sulfur (S) K_{α} /Pb M_{α} . In the As/Pb case, Pb can be measured from the Pb L_{β} line, and As can be measured from either the As K_{α} or the As K_{β} line; in this way the interference can be corrected. If the As K_{β} line is used, sensitivity will be decreased by a factor of two to five times because it is a less intense line than the As K_{α} line. If the As K_{α} line is used in the presence of Pb, mathematical corrections within the instrument software can be used to subtract out the Pb interference. However, because of the limits of mathematical corrections, As concentrations cannot be efficiently calculated for samples with Pb:As ratios of 10:1 or more. This high ratio of Pb to As may result in no As being reported regardless of the actual concentration present.

No instrument can fully compensate for this interference. It is important for an operator to understand this limitation of FPXRF instruments and consult with the manufacturer of the FPXRF instrument to evaluate options to minimize this limitation. The operator's decision will be based on action levels for metals in soil established for the site, matrix effects, capabilities of the instrument, data quality objectives, and the ratio of lead to arsenic known to be present at the site. If a site is encountered that contains lead at concentrations greater than ten times the concentration of arsenic it is advisable that all critical soil samples be sent off site for confirmatory analysis by an EPA-approved method.

4.8 If SSCS are used to calibrate an FPXRF instrument, the samples collected must be representative of the site under investigation. Representative soil sampling ensures that a sample or group of samples accurately reflects the concentrations of the contaminants of concern at a given time and location. Analytical results for representative samples reflect variations in the presence and concentration ranges of contaminants throughout a site. Variables affecting sample representativeness include differences in soil type, contaminant concentration variability, sample collection and preparation variability, and analytical variability, all of which should be minimized as much as possible.

4.9 Soil physical and chemical effects may be corrected using SSCS that have been analyzed by inductively coupled plasma (ICP) or atomic absorption (AA) methods. However, a major source of error can be introduced if these samples are not representative of the site or if the analytical error is large. Another concern is the type of digestion procedure used to prepare the soil samples for the reference analysis. Analytical results for the confirmatory method will vary depending on whether a partial digestion procedure, such as SW-846 Method 3050, or a total digestion procedure, such as Method 3052 is used. It is known that depending on the nature of the soil or sediment, Method 3050 will achieve differing extraction efficiencies for different analytes of interest. The confirmatory method should meet the project data quality objectives.

XRF measures the total concentration of an element; therefore, to achieve the greatest comparability of this method with the reference method (reduced bias), a total digestion procedure should be used for sample preparation. However, in the study used to generate the performance data for this method, the confirmatory method used was Method 3050, and the FPXRF data

compared very well with regression correlation coefficients (r^2 often exceeding 0.95, except for barium and chromium. See Table 9 in Section 17.0). The critical factor is that the digestion procedure and analytical reference method used should meet the data quality objectives (DQOs) of the project and match the method used for confirmation analysis.

4.10 Ambient temperature changes can affect the gain of the amplifiers producing instrument drift. Gain or drift is primarily a function of the electronics (amplifier or preamplifier) and not the detector as most instrument detectors are cooled to a constant temperature. Most FPXRF instruments have a built-in automatic gain control. If the automatic gain control is allowed to make periodic adjustments, the instrument will compensate for the influence of temperature changes on its energy scale. If the FPXRF instrument has an automatic gain control function, the operator will not have to adjust the instrument's gain unless an error message appears. If an error message appears, the operator should follow the manufacturer's procedures for troubleshooting the problem. Often, this involves performing a new energy calibration. The performance of an energy calibration check to assess drift is a quality control measure discussed in Section 9.2.

If the operator is instructed by the manufacturer to manually conduct a gain check because of increasing or decreasing ambient temperature, it is standard to perform a gain check after every 10 to 20 sample measurements or once an hour whichever is more frequent. It is also suggested that a gain check be performed if the temperature fluctuates more than 10 to 20°F. The operator should follow the manufacturer's recommendations for gain check frequency.

5.0 SAFETY

5.1 Proper training for the safe operation of the instrument and radiation training should be completed by the analyst prior to analysis. Radiation safety for each specific instrument can be found in the operators manual. Protective shielding should never be removed by the analyst or any personnel other than the manufacturer. The analyst should be aware of the local state and national regulations that pertain to the use of radiation-producing equipment and radioactive materials with which compliance is required. Licenses for radioactive materials are of two types; (1) general license which is usually provided by the manufacturer for receiving, acquiring, owning, possessing, using, and transferring radioactive material incorporated in a device or equipment, and (2) specific license which is issued to named persons for the operation of radioactive instruments as required by local state agencies. There should be a person appointed within the organization that is solely responsible for properly instructing all personnel, maintaining inspection records, and monitoring x-ray equipment at regular intervals. A copy of the radioactive material licenses and leak tests should be present with the instrument at all times and available to local and national authorities upon request. X-ray tubes do not require radioactive material licenses or leak tests, but do require approvals and licenses which vary from state to state. In addition, fail-safe x-ray warning lights should be illuminated whenever an x-ray tube is energized. Provisions listed above concerning radiation safety regulations, shielding, training, and responsible personnel apply to x-ray tubes just as to radioactive sources. In addition, a log of the times and operating conditions should be kept whenever an x-ray tube is energized. Finally, an additional hazard present with x-ray tubes is the danger of electric shock from the high voltage supply. The danger of electric shock is as substantial as the danger from radiation but is often overlooked because of its familiarity.

5.2 Radiation monitoring equipment should be used with the handling of the instrument. The operator and the surrounding environment should be monitored continually for analyst exposure to radiation. Thermal luminescent detectors (TLD) in the form of badges and rings are used to monitor operator radiation exposure. The TLDs should be worn in the area of most frequent exposure. The maximum permissible whole-body dose from occupational exposure is 5

Roentgen Equivalent Man (REM) per year. Possible exposure pathways for radiation to enter the body are ingestion, inhaling, and absorption. The best precaution to prevent radiation exposure is distance and shielding.

5.3 Refer to Chapter Three for guidance on some proper safety protocols.

6.0 EQUIPMENT AND SUPPLIES

6.1 FPXRF Spectrometer: An FPXRF spectrometer consists of four major components: (1) a source that provides x-rays; (2) a sample presentation device; (3) a detector that converts x-ray-generated photons emitted from the sample into measurable electronic signals; and (4) a data processing unit that contains an emission or fluorescence energy analyzer, such as an MCA, that processes the signals into an x-ray energy spectrum from which elemental concentrations in the sample may be calculated, and a data display and storage system. These components and additional, optional items, are discussed below.

6.1.1 Excitation Sources: Most FPXRF instruments use sealed radioisotope sources to produce x-rays in order to irradiate samples. The FPXRF instrument may contain between one and three radioisotope sources. Common radioisotope sources used for analysis for metals in soils are iron (Fe)-55, cadmium (Cd)-109, americium (Am)-241, and curium (Cm)-244. These sources may be contained in a probe along with a window and the detector; the probe is connected to a data reduction and handling system by means of a flexible cable. Alternatively, the sources, window, and detector may be included in the same unit as the data reduction and handling system.

The relative strength of the radioisotope sources is measured in units of millicuries (mCi). All other components of the FPXRF system being equal, the stronger the source, the greater the sensitivity and precision of a given instrument. Radioisotope sources undergo constant decay. In fact, it is this decay process that emits the primary x-rays used to excite samples for FPXRF analysis. The decay of radioisotopes is measured in "half-lives." The half-life of a radioisotope is defined as the length of time required to reduce the radioisotopes strength or activity by half. Developers of FPXRF technologies recommend source replacement at regular intervals based on the source's half-life. The characteristic x-rays emitted from each of the different sources have energies capable of exciting a certain range of analytes in a sample. Table 2 summarizes the characteristics of four common radioisotope sources.

X-ray tubes have higher radiation output, no intrinsic lifetime limit, produce constant output over their lifetime, and do not have the disposal problems of radioactive sources but are just now appearing in FPXRF instruments. An electrically-excited x-ray tube operates by bombarding an anode with electrons accelerated by a high voltage. The electrons gain an energy in electron volts equal to the accelerating voltage and can excite atomic transitions in the anode, which then produces characteristic x-rays. These characteristic x-rays are emitted through a window which contains the vacuum required for the electron acceleration. An important difference between x-ray tubes and radioactive sources is that the electrons which bombard the anode also produce a continuum of x-rays across a broad range of energies in addition to the characteristic x-rays. This continuum is weak compared to the characteristic x-rays but can provide substantial excitation since it covers a broad energy range. It has the undesired property of producing background in the spectrum near the analyte x-ray lines when it is scattered by the sample. For this reason a filter is often used between the x-ray tube and the sample to suppress the continuum radiation while passing the characteristic

x-rays from the anode. This filter is sometimes incorporated into the window of the x-ray tube. The choice of accelerating voltage is governed by the anode material, since the electrons must have sufficient energy to excite the anode, which requires a voltage greater than the absorption edge of the anode material. The anode is most efficiently excited by voltages 2 to 2.5 times the edge energy (most x-rays per unit power to the tube), although voltages as low as 1.5 times the absorption edge energy will work. The characteristic x-rays emitted by the anode are capable of exciting a range of elements in the sample just as with a radioactive source. Table 3 gives the recommended operating voltages and the sample elements excited for some common anodes.

6.1.2 Sample Presentation Device: FPXRF instruments can be operated in two modes: in situ and intrusive. If operated in the in situ mode, the probe window is placed in direct contact with the soil surface to be analyzed. When an FPXRF instrument is operated in the intrusive mode, a soil or sediment sample must be collected, prepared, and placed in a sample cup. For most FPXRF instruments operated in the intrusive mode, the probe is rotated so that the window faces upward. A protective sample cover is placed over the window, and the sample cup is placed on top of the window inside the protective sample cover for analysis.

6.1.3 Detectors: The detectors in the FPXRF instruments can be either solid-state detectors or gas-filled, proportional counter detectors. Common solid-state detectors include mercuric iodide (HgI_2), silicon pin diode and lithium-drifted silicon $\text{Si}(\text{Li})$. The HgI_2 detector is operated at a moderately subambient temperature controlled by a low power thermoelectric cooler. The silicon pin diode detector also is cooled via the thermoelectric Peltier effect. The $\text{Si}(\text{Li})$ detector must be cooled to at least -90°C either with liquid nitrogen or by thermoelectric cooling via the Peltier effect. Instruments with a $\text{Si}(\text{Li})$ detector have an internal liquid nitrogen dewar with a capacity of 0.5 to 1.0 liter. Proportional counter detectors are rugged and lightweight, which are important features of a field portable detector. However, the resolution of a proportional counter detector is not as good as that of a solid-state detector. The energy resolution of a detector for characteristic x-rays is usually expressed in terms of full width at half-maximum (FWHM) height of the manganese K_α peak at 5.89 keV. The typical resolutions of the above mentioned detectors are as follows: HgI_2 -270 eV; silicon pin diode-250 eV; $\text{Si}(\text{Li})$ -170 eV; and gas-filled, proportional counter-750 eV.

During operation of a solid-state detector, an x-ray photon strikes a biased, solid-state crystal and loses energy in the crystal by producing electron-hole pairs. The electric charge produced is collected and provides a current pulse that is directly proportional to the energy of the x-ray photon absorbed by the crystal of the detector. A gas-filled, proportional counter detector is an ionization chamber filled with a mixture of noble and other gases. An x-ray photon entering the chamber ionizes the gas atoms. The electric charge produced is collected and provides an electric signal that is directly proportional to the energy of the x-ray photon absorbed by the gas in the detector.

6.1.4 Data Processing Units: The key component in the data processing unit of an FPXRF instrument is the MCA. The MCA receives pulses from the detector and sorts them by their amplitudes (energy level). The MCA counts pulses per second to determine the height of the peak in a spectrum, which is indicative of the target analyte's concentration. The spectrum of element peaks are built on the MCA. The MCAs in FPXRF instruments have from 256 to 2,048 channels. The concentrations of target analytes are usually shown in parts per million on a liquid crystal display (LCD) in the instrument. FPXRF instruments can store both spectra and from 100 to 500 sets of numerical analytical results. Most FPXRF

instruments are menu-driven from software built into the units or from PCs. Once the data-storage memory of an FPXRF unit is full, data can be downloaded by means of an RS-232 port and cable to a PC.

6.2 Spare battery chargers.

6.3 Polyethylene sample cups: 31 millimeters (mm) to 40 mm in diameter with collar, or equivalent (appropriate for FPXRF instrument).

6.4 X-ray window film: Mylar™, Kapton™, Spectrolene™, polypropylene, or equivalent; 2.5 to 6.0 micrometers (μm) thick.

6.5 Mortar and pestle: glass, agate, or aluminum oxide; for grinding soil and sediment samples.

6.6 Containers: glass or plastic to store samples.

6.7 Sieves: 60-mesh (0.25 mm), stainless-steel, Nylon, or equivalent for preparing soil and sediment samples.

6.8 Trowels: for smoothing soil surfaces and collecting soil samples.

6.9 Plastic bags: used for collection and homogenization of soil samples.

6.10 Drying oven: standard convection or toaster oven, for soil and sediment samples that require drying.

7.0 REAGENTS AND STANDARDS

7.1 Pure Element Standards: Each pure, single-element standard is intended to produce strong characteristic x-ray peaks of the element of interest only. Other elements present must not contribute to the fluorescence spectrum. A set of pure element standards for commonly sought analytes is supplied by the instrument manufacturer, if required for the instrument; not all instruments require the pure element standards. The standards are used to set the region of interest (ROI) for each element. They also can be used as energy calibration and resolution check samples.

7.2 Site-specific Calibration Standards: Instruments that employ fundamental parameters (FP) or similar mathematical models in minimizing matrix effects may not require SSCS. If the FP calibration model is to be optimized or if empirical calibration is necessary, then SSCSs must be collected, prepared, and analyzed.

7.2.1 The SSCS must be representative of the matrix to be analyzed by FPXRF. These samples must be well homogenized. A minimum of ten samples spanning the concentration ranges of the analytes of interest and of the interfering elements must be obtained from the site. A sample size of 4 to 8 ounces is recommended, and standard glass sampling jars should be used.

7.2.2 Each sample should be oven-dried for 2 to 4 hours at a temperature of less than 150°C. If mercury is to be analyzed, a separate sample portion must remain undried, as heating may volatilize the mercury. When the sample is dry, all large, organic debris and

nonrepresentative material, such as twigs, leaves, roots, insects, asphalt, and rock should be removed. The sample should be ground with a mortar and pestle and passed through a 60-mesh sieve. Only the coarse rock fraction should remain on the screen.

7.2.3 The sample should be homogenized by using a riffle splitter or by placing 150 to 200 grams of the dried, sieved sample on a piece of kraft or butcher paper about 1.5 by 1.5 feet in size. Each corner of the paper should be lifted alternately, rolling the soil over on itself and toward the opposite corner. The soil should be rolled on itself 20 times. Approximately 5 grams of the sample should then be removed and placed in a sample cup for FPXRF analysis. The rest of the prepared sample should be sent off site for ICP or AA analysis. The method use for confirmatory analysis should meet the data quality objectives of the project.

7.3 Blank Samples: The blank samples should be from a "clean" quartz or silicon dioxide matrix that is free of any analytes at concentrations above the method detection limits. These samples are used to monitor for cross-contamination and laboratory-induced contaminants or interferences.

7.4 Standard Reference Materials: Standard reference materials (SRM) are standards containing certified amounts of metals in soil or sediment. These standards are used for accuracy and performance checks of FPXRF analyses. SRMs can be obtained from the National Institute of Standards and Technology (NIST), the U.S. Geological Survey (USGS), the Canadian National Research Council, and the national bureau of standards in foreign nations. Pertinent NIST SRMs for FPXRF analysis include 2704, Buffalo River Sediment; 2709, San Joaquin Soil; and 2710 and 2711, Montana Soil. These SRMs contain soil or sediment from actual sites that has been analyzed using independent inorganic analytical methods by many different laboratories.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

Sample handling and preservation procedures used in FPXRF analyses should follow the guidelines in Chapter Three, Inorganic Analytes.

9.0 QUALITY CONTROL

9.1 Refer to Chapter One for additional guidance on quality assurance protocols. All field data sheets and quality control data should be maintained for reference or inspection.

9.2 Energy Calibration Check: To determine whether an FPXRF instrument is operating within resolution and stability tolerances, an energy calibration check should be run. The energy calibration check determines whether the characteristic x-ray lines are shifting, which would indicate drift within the instrument. As discussed in Section 4.10, this check also serves as a gain check in the event that ambient temperatures are fluctuating greatly (> 10 to 20°F).

The energy calibration check should be run at a frequency consistent with manufacturers recommendations. Generally, this would be at the beginning of each working day, after the batteries are changed or the instrument is shut off, at the end of each working day, and at any other time when the instrument operator believes that drift is occurring during analysis. A pure element such as iron, manganese, copper, or lead is often used for the energy calibration check. A manufacturer-recommended count time per source should be used for the check.

9.2.1 The instrument manufacturer's manual specifies the channel or kiloelectron volt level at which a pure element peak should appear and the expected intensity of the peak.

The intensity and channel number of the pure element as measured using the radioactive source should be checked and compared to the manufacturer's recommendation. If the energy calibration check does not meet the manufacturer's criteria, then the pure element sample should be repositioned and reanalyzed. If the criteria are still not met, then an energy calibration should be performed as described in the manufacturer's manual. With some FPXRF instruments, once a spectrum is acquired from the energy calibration check, the peak can be optimized and realigned to the manufacturer's specifications using their software.

9.3 Blank Samples: Two types of blank samples should be analyzed for FPXRF analysis: instrument blanks and method blanks. An instrument blank is used to verify that no contamination exists in the spectrometer or on the probe window.

9.3.1 The instrument blank can be silicon dioxide, a Teflon block, a quartz block, "clean" sand, or lithium carbonate. This instrument blank should be analyzed on each working day before and after analyses are conducted and once per every twenty samples. An instrument blank should also be analyzed whenever contamination is suspected by the analyst. The frequency of analysis will vary with the data quality objectives of the project. A manufacturer-recommended count time per source should be used for the blank analysis. No element concentrations above the method detection limits should be found in the instrument blank. If concentrations exceed these limits, then the probe window and the check sample should be checked for contamination. If contamination is not a problem, then the instrument must be "zeroed" by following the manufacturer's instructions.

9.3.2 A method blank is used to monitor for laboratory-induced contaminants or interferences. The method blank can be "clean" silica sand or lithium carbonate that undergoes the same preparation procedure as the samples. A method blank must be analyzed at least daily. The frequency of analysis will depend on the data quality objectives of the project. To be acceptable, a method blank must not contain any analyte at a concentration above its method detection limit. If an analyte's concentration exceeds its method detection limit, the cause of the problem must be identified, and all samples analyzed with the method blank must be reanalyzed.

9.4 Calibration Verification Checks: A calibration verification check sample is used to check the accuracy of the instrument and to assess the stability and consistency of the analysis for the analytes of interest. A check sample should be analyzed at the beginning of each working day, during active sample analyses, and at the end of each working day. The frequency of calibration checks during active analysis will depend on the data quality objectives of the project. The check sample should be a well characterized soil sample from the site that is representative of site samples in terms of particle size and degree of homogeneity and that contains contaminants at concentrations near the action levels. If a site-specific sample is not available, then an NIST or other SRM that contains the analytes of interest can be used to verify the accuracy of the instrument. The measured value for each target analyte should be within ± 20 percent (%D) of the true value for the calibration verification check to be acceptable. If a measured value falls outside this range, then the check sample should be reanalyzed. If the value continues to fall outside the acceptance range, the instrument should be recalibrated, and the batch of samples analyzed before the unacceptable calibration verification check must be reanalyzed.

9.5 Precision Measurements: The precision of the method is monitored by analyzing a sample with low, moderate, or high concentrations of target analytes. The frequency of precision measurements will depend on the data quality objectives for the data. A minimum of one precision sample should be run per day. Each precision sample should be analyzed 7 times in replicate. It

is recommended that precision measurements be obtained for samples with varying concentration ranges to assess the effect of concentration on method precision. Determining method precision for analytes at concentrations near the site action levels can be extremely important if the FPXRF results are to be used in an enforcement action; therefore, selection of at least one sample with target analyte concentrations at or near the site action levels or levels of concern is recommended. A precision sample is analyzed by the instrument for the same field analysis time as used for other project samples. The relative standard deviation (RSD) of the sample mean is used to assess method precision. For FPXRF data to be considered adequately precise, the RSD should not be greater than 20 percent with the exception of chromium. RSD values for chromium should not be greater than 30 percent.

The equation for calculating RSD is as follows:

$$\text{RSD} = (\text{SD}/\text{Mean Concentration}) \times 100$$

where:

RSD	=	Relative standard deviation for the precision measurement for the analyte
SD	=	Standard deviation of the concentration for the analyte
Mean Concentration	=	Mean concentration for the analyte

The precision or reproducibility of a measurement will improve with increasing count time, however, increasing the count time by a factor of 4 will provide only 2 times better precision, so there is a point of diminishing return. Increasing the count time also improves the detection limit, but decreases sample throughput.

9.6 Detection Limits: Results for replicate analyses of a low-concentration sample, SSCS, or SRM can be used to generate an average site-specific method detection and quantitation limits. In this case, the method detection limit is defined as 3 times the standard deviation of the results for the low-concentration samples and the method quantitation limit is defined as 10 times the standard deviation of the same results. Another means of determining method detection and quantitation limits involves use of counting statistics. In FPXRF analysis, the standard deviation from counting statistics is defined as $\text{SD} = (N)^{1/2}$, where SD is the standard deviation for a target analyte peak and N is the net counts for the peak of the analyte of interest (i.e., gross counts minus background under the peak). Three times this standard deviation would be the method detection limit and 10 times this standard deviation would be the method quantitation limit. If both of the above mentioned approaches are used to calculate method detection limits, the larger of the standard deviations should be used to provide the more conservative detection limits.

This SD based detection limit criteria must be used by the operator to evaluate each measurement for its useability. A measurement above the average calculated or manufacturer's detection limit, but smaller than three times its associated SD, should not be used as a quantitative measurement. Conversely, if the measurement is below the average calculated or manufacturer's detection limit, but greater than three times its associated SD. It should be coded as an estimated value.

9.7 Confirmatory Samples: The comparability of the FPXRF analysis is determined by submitting FPXRF-analyzed samples for analysis at a laboratory. The method of confirmatory analysis must meet the project and XRF measurement data quality objectives. The confirmatory samples must be splits of the well homogenized sample material. In some cases the prepared

sample cups can be submitted. A minimum of 1 sample for each 20 FPXRF-analyzed samples should be submitted for confirmatory analysis. This frequency will depend on data quality objectives. The confirmatory analyses can also be used to verify the quality of the FPXRF data. The confirmatory samples should be selected from the lower, middle, and upper range of concentrations measured by the FPXRF. They should also include samples with analyte concentrations at or near the site action levels. The results of the confirmatory analysis and FPXRF analyses should be evaluated with a least squares linear regression analysis. If the measured concentrations span more than one order of magnitude, the data should be log-transformed to standardize variance which is proportional to the magnitude of measurement. The correlation coefficient (r^2) for the results should be 0.7, or greater for the FPXRF data to be considered screening level data. If the r^2 is 0.9 or greater and inferential statistics indicate the FPXRF data and the confirmatory data are statistically equivalent at a 99 percent confidence level, the data could potentially meet definitive level data criteria.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Instrument Calibration: Instrument calibration procedures vary among FPXRF instruments. Users of this method should follow the calibration procedures outlined in the operator's manual for each specific FPXRF instrument. Generally, however, three types of calibration procedures exist for FPXRF instruments: FP calibration, empirical calibration, and the Compton peak ratio or normalization method. These three types of calibration are discussed below.

10.2 Fundamental Parameters Calibration: FP calibration procedures are extremely variable. An FP calibration provides the analyst with a "standardless" calibration. The advantages of FP calibrations over empirical calibrations include the following:

- No previously collected site-specific samples are required, although site-specific samples with confirmed and validated analytical results for all elements present could be used.
- Cost is reduced because fewer confirmatory laboratory results or calibration standards are required.

However, the analyst should be aware of the limitations imposed on FP calibration by particle size and matrix effects. These limitations can be minimized by adhering to the preparation procedure described in Section 7.2. The two FP calibration processes discussed below are based on an effective energy FP routine and a back scatter with FP (BFP) routine. Each FPXRF FP calibration process is based on a different iterative algorithmic method. The calibration procedure for each routine is explained in detail in the manufacturer's user manual for each FPXRF instrument; in addition, training courses are offered for each instrument.

10.2.1 Effective Energy FP Calibration: The effective energy FP calibration is performed by the manufacturer before an instrument is sent to the analyst. Although SSCS can be used, the calibration relies on pure element standards or SRMs such as those obtained from NIST for the FP calibration. The effective energy routine relies on the spectrometer response to pure elements and FP iterative algorithms to compensate for various matrix effects.

Alpha coefficients are calculated using a variation of the Sherman equation, which calculates theoretical intensities from the measurement of pure element samples. These coefficients indicate the quantitative effect of each matrix element on an analyte's measured

x-ray intensity. Next, the Lachance Traill algorithm is solved as a set of simultaneous equations based on the theoretical intensities. The alpha coefficients are then downloaded into the specific instrument.

The working effective energy FP calibration curve must be verified before sample analysis begins on each working day, after every 20 samples are analyzed, and at the end of sampling. This verification is performed by analyzing either an NIST SRM or an SSCS that is representative of the site-specific samples. This SRM or SSCS serves as a calibration check. A manufacturer-recommended count time per source should be used for the calibration check. The analyst must then adjust the y-intercept and slope of the calibration curve to best fit the known concentrations of target analytes in the SRM or SSCS.

A percent difference (%D) is then calculated for each target analyte. The %D should be within ± 20 percent of the certified value for each analyte. If the %D falls outside this acceptance range, then the calibration curve should be adjusted by varying the slope of the line or the y-intercept value for the analyte. The SRM or SSCS is reanalyzed until the %D falls within ± 20 percent. The group of 20 samples analyzed before an out-of-control calibration check should be reanalyzed.

The equation to calibrate %D is as follows:

$$\%D = ((C_s - C_k) / C_k) \times 100$$

where:

%D = Percent difference

C_k = Certified concentration of standard sample

C_s = Measured concentration of standard sample

10.2.2 BFP Calibration: BFP calibration relies on the ability of the liquid nitrogen-cooled, Si(Li) solid-state detector to separate the coherent (Compton) and incoherent (Rayleigh) backscatter peaks of primary radiation. These peak intensities are known to be a function of sample composition, and the ratio of the Compton to Rayleigh peak is a function of the mass absorption of the sample. The calibration procedure is explained in detail in the instrument manufacturer's manual. Following is a general description of the BFP calibration procedure.

The concentrations of all detected and quantified elements are entered into the computer software system. Certified element results for an NIST SRM or confirmed and validated results for an SSCS can be used. In addition, the concentrations of oxygen and silicon must be entered; these two concentrations are not found in standard metals analyses. The manufacturer provides silicon and oxygen concentrations for typical soil types. Pure element standards are then analyzed using a manufacturer-recommended count time per source. The results are used to calculate correction factors in order to adjust for spectrum overlap of elements.

The working BFP calibration curve must be verified before sample analysis begins on each working day, after every 20 samples are analyzed, and at the end of the analysis. This verification is performed by analyzing either an NIST SRM or an SSCS that is representative of the site-specific samples. This SRM or SSCS serves as a calibration check. The standard sample is analyzed using a manufacturer-recommended count time per source to check the

calibration curve. The analyst must then adjust the y-intercept and slope of the calibration curve to best fit the known concentrations of target analytes in the SRM or SSCS.

A %D is then calculated for each target analyte. The %D should fall within ± 20 percent of the certified value for each analyte. If the %D falls outside this acceptance range, then the calibration curve should be adjusted by varying the slope of the line the y-intercept value for the analyte. The standard sample is reanalyzed until the %D falls within ± 20 percent. The group of 20 samples analyzed before an out-of-control calibration check should be reanalyzed.

10.3 Empirical Calibration: An empirical calibration can be performed with SSCS, site-typical standards, or standards prepared from metal oxides. A discussion of SSCS is included in Section 7.2; if no previously characterized samples exist for a specific site, site-typical standards can be used. Site-typical standards may be selected from commercially available characterized soils or from SSCS prepared for another site. The site-typical standards should closely approximate the site's soil matrix with respect to particle size distribution, mineralogy, and contaminant analytes. If neither SSCS nor site-typical standards are available, it is possible to make gravimetric standards by adding metal oxides to a "clean" sand or silicon dioxide matrix that simulates soil. Metal oxides can be purchased from various chemical vendors. If standards are made on site, a balance capable of weighing items to at least two decimal places is required. Concentrated ICP or AA standard solutions can also be used to make standards. These solutions are available in concentrations of 10,000 parts per million, thus only small volumes have to be added to the soil.

An empirical calibration using SSCS involves analysis of SSCS by the FPXRF instrument and by a conventional analytical method such as ICP or AA. A total acid digestion procedure should be used by the laboratory for sample preparation. Generally, a minimum of 10 and a maximum of 30 well characterized SSCS, site-typical standards, or prepared metal oxide standards are required to perform an adequate empirical calibration. The number of required standards depends on the number of analytes of interest and interfering elements. Theoretically, an empirical calibration with SSCS should provide the most accurate data for a site because the calibration compensates for site-specific matrix effects.

The first step in an empirical calibration is to analyze the pure element standards for the elements of interest. This enables the instrument to set channel limits for each element for spectral deconvolution. Next the SSCS, site-typical standards, or prepared metal oxide standards are analyzed using a count time of 200 seconds per source or a count time recommended by the manufacturer. This will produce a spectrum and net intensity of each analyte in each standard. The analyte concentrations for each standard are then entered into the instrument software; these concentrations are those obtained from the laboratory, the certified results, or the gravimetrically determined concentrations of the prepared standards. This gives the instrument analyte values to regress against corresponding intensities during the modeling stage. The regression equation correlates the concentrations of an analyte with its net intensity.

The calibration equation is developed using a least squares fit regression analysis. After the regression terms to be used in the equation are defined, a mathematical equation can be developed to calculate the analyte concentration in an unknown sample. In some FPXRF instruments, the software of the instrument calculates the regression equation. The software uses calculated intercept and slope values to form a multiterm equation. In conjunction with the software in the instrument, the operator can adjust the multiterm equation to minimize interelement interferences and optimize the intensity calibration curve.

It is possible to define up to six linear or nonlinear terms in the regression equation. Terms can be added and deleted to optimize the equation. The goal is to produce an equation with the smallest regression error and the highest correlation coefficient. These values are automatically computed by the software as the regression terms are added, deleted, or modified. It is also possible to delete data points from the regression line if these points are significant outliers or if they are heavily weighing the data. Once the regression equation has been selected for an analyte, the equation can be entered into the software for quantitation of analytes in subsequent samples. For an empirical calibration to be acceptable, the regression equation for a specific analyte should have a correlation coefficient of 0.98 or greater or meet the DQOs of the project.

In an empirical calibration, one must apply the DQOs of the project and ascertain critical or action levels for the analytes of interest. It is within these concentration ranges or around these action levels that the FPXRF instrument should be calibrated most accurately. It may not be possible to develop a good regression equation over several orders of analyte concentration.

10.4 Compton Normalization Method: The Compton normalization method is based on analysis of a single, certified standard and normalization for the Compton peak. The Compton peak is produced from incoherent backscattering of x-ray radiation from the excitation source and is present in the spectrum of every sample. The Compton peak intensity changes with differing matrices. Generally, matrices dominated by lighter elements produce a larger Compton peak, and those dominated by heavier elements produce a smaller Compton peak. Normalizing to the Compton peak can reduce problems with varying matrix effects among samples. Compton normalization is similar to the use of internal standards in organics analysis. The Compton normalization method may not be effective when analyte concentrations exceed a few percent.

The certified standard used for this type of calibration could be an NIST SRM such as 2710 or 2711. The SRM must be a matrix similar to the samples and must contain the analytes of interests at concentrations near those expected in the samples. First, a response factor has to be determined for each analyte. This factor is calculated by dividing the net peak intensity by the analyte concentration. The net peak intensity is gross intensity corrected for baseline interference. Concentrations of analytes in samples are then determined by multiplying the baseline corrected analyte signal intensity by the normalization factor and by the response factor. The normalization factor is the quotient of the baseline corrected Compton K_{α} peak intensity of the SRM divided by that of the samples. Depending on the FPXRF instrument used, these calculations may be done manually or by the instrument software.

11.0 PROCEDURE

11.1 Operation of the various FPXRF instruments will vary according to the manufacturers' protocols. Before operating any FPXRF instrument, one should consult the manufacturer's manual. Most manufacturers recommend that their instruments be allowed to warm up for 15 to 30 minutes before analysis of samples. This will help alleviate drift or energy calibration problems later on in analysis.

11.2 Each FPXRF instrument should be operated according to the manufacturer's recommendations. There are two modes in which FPXRF instruments can be operated: in situ and intrusive. The in situ mode involves analysis of an undisturbed soil sediment or sample. Intrusive analysis involves collection and preparation of a soil or sediment sample before analysis. Some FPXRF instruments can operate in both modes of analysis, while others are designed to operate in only one mode. The two modes of analysis are discussed below.

11.3 For *in situ* analysis, one requirement is that any large or nonrepresentative debris be removed from the soil surface before analysis. This debris includes rocks, pebbles, leaves, vegetation, roots, and concrete. Another requirement is that the soil surface be as smooth as possible so that the probe window will have good contact with the surface. This may require some leveling of the surface with a stainless-steel trowel. During the study conducted to provide data for this method, this modest amount of sample preparation was found to take less than 5 minutes per sample location. The last requirement is that the soil or sediment not be saturated with water. Manufacturers state that their FPXRF instruments will perform adequately for soils with moisture contents of 5 to 20 percent but will not perform well for saturated soils, especially if ponded water exists on the surface. Another recommended technique for *in situ* analysis is to tamp the soil to increase soil density and compactness for better repeatability and representativeness. This condition is especially important for heavy element analysis, such as barium. Source count times for *in situ* analysis usually range from 30 to 120 seconds, but source count times will vary among instruments and depending on required detection limits.

11.4 For intrusive analysis of surface or sediment, it is recommended that a sample be collected from a 4- by 4-inch square that is 1 inch deep. This will produce a soil sample of approximately 375 grams or 250 cm³, which is enough soil to fill an 8-ounce jar. The sample should be homogenized, dried, and ground before analysis. The sample can be homogenized before or after drying. The homogenization technique to be used after drying is discussed in Section 4.2. If the sample is homogenized before drying, it should be thoroughly mixed in a beaker or similar container, or if the sample is moist and has a high clay content, it can be kneaded in a plastic bag. One way to monitor homogenization when the sample is kneaded in a plastic bag is to add sodium fluorescein dye to the sample. After the moist sample has been homogenized, it is examined under an ultraviolet light to assess the distribution of sodium fluorescein throughout the sample. If the fluorescent dye is evenly distributed in the sample, homogenization is considered complete; if the dye is not evenly distributed, mixing should continue until the sample has been thoroughly homogenized. During the study conducted to provide data for this method, the homogenization procedure using the fluorescein dye required 3 to 5 minutes per sample. As demonstrated in Sections 13.5 and 13.7, homogenization has the greatest impact on the reduction of sampling variability. It produces little or no contamination. Often, it can be used without the more labor intensive steps of drying, grinding, and sieving given in Sections 11.5 and 11.6. Of course, to achieve the best data quality possible all four steps must be followed.

11.5 Once the soil or sediment sample has been homogenized, it should be dried. This can be accomplished with a toaster oven or convection oven. A small aliquot of the sample (20 to 50 grams) is placed in a suitable container for drying. The sample should be dried for 2 to 4 hours in the convection or toaster oven at a temperature not greater than 150°C. Microwave drying is not a recommended procedure. Field studies have shown that microwave drying can increase variability between the FPXRF data and confirmatory analysis. High levels of metals in a sample can cause arcing in the microwave oven, and sometimes slag forms in the sample. Microwave oven drying can also melt plastic containers used to hold the sample.

11.6 The homogenized dried sample material should be ground with a mortar and pestle and passed through a 60-mesh sieve to achieve a uniform particle size. Sample grinding should continue until at least 90 percent of the original sample passes through the sieve. The grinding step normally takes an average of 10 minutes per sample. An aliquot of the sieved sample should then be placed in a 31.0-mm polyethylene sample cup (or equivalent) for analysis. The sample cup should be one-half to three-quarters full at a minimum. The sample cup should be covered with a 2.5 µm Mylar (or equivalent) film for analysis. The rest of the soil sample should be placed in a jar, labeled, and archived for possible confirmation analysis. All equipment including the mortar, pestle,

and sieves must be thoroughly cleaned so that any cross-contamination is below the MDLs of the procedure or DQOs of the analysis.

12.0 DATA ANALYSIS AND CALCULATIONS

Most FPXRF instruments have software capable of storing all analytical results and spectra. The results are displayed in parts per million and can be downloaded to a PC, which can provide a hard copy printout. Individual measurements that are smaller than three times their associated SD should not be used for quantitation.

13.0 METHOD PERFORMANCE

13.1 This section discusses four performance factors, field-based method detection limits, precision, accuracy, and comparability to EPA-approved methods. The numbers presented in Tables 4 through 9 were generated from data obtained from six FPXRF instruments. The soil samples analyzed by the six FPXRF instruments were collected from two sites in the United States. The soil samples contained several of the target analytes at concentrations ranging from nondetect to tens of thousands of mg/kg.

13.2 The six FPXRF instruments included the TN 9000 and TN Lead Analyzer manufactured by TN Spectrace; the X-MET 920 with a SiLi detector and X-MET 920 with a gas-filled proportional detector manufactured by Metorex, Inc.; the XL Spectrum Analyzer manufactured by Nilon; and the MAP Spectrum Analyzer manufactured by Scitec. The TN 9000 and TN Lead Analyzer both have a HgI₂ detector. The TN 9000 utilized an Fe-55, Cd-109, and Am-241 source. The TN Lead Analyzer had only a Cd-109 source. The X-Met 920 with the SiLi detector had a Cd-109 and Am-241 source. The X-MET 920 with the gas-filled proportional detector had only a Cd-109 source. The XL Spectrum Analyzer utilized a silicon pin-diode detector and a Cd-109 source. The MAP Spectrum Analyzer utilized a solid-state silicon detector and a Cd-109 source.

13.3 All data presented in Tables 4 through 9 were generated using the following calibrations and source count times. The TN 9000 and TN Lead Analyzer were calibrated using fundamental parameters using NIST SRM 2710 as a calibration check sample. The TN 9000 was operated using 100, 60, and 60 second count times for the Cd-109, Fe-55, and Am-241 sources, respectively. The TN Lead analyzer was operated using a 60 second count time for the Cd-109 source. The X-MET 920 with the Si(Li) detector was calibrated using fundamental parameters and one well characterized site-specific soil standard as a calibration check. It used 140 and 100 second count times for the Cd-109 and Am-241 sources, respectively. The X-MET 920 with the gas-filled proportional detector was calibrated empirically using between 10 and 20 well characterized site-specific soil standards. It used 120 second times for the Cd-109 source. The XL Spectrum Analyzer utilized NIST SRM 2710 for calibration and the Compton peak normalization procedure for quantitation based on 60 second count times for the Cd-109 source. The MAP Spectrum Analyzer was internally calibrated by the manufacturer. The calibration was checked using a well-characterized site-specific soil standard. It used 240 second times for the Cd-109 source.

13.4 Field-Based Method Detection Limits: The field-based method detection limits are presented in Table 4. The field-based method detection limits were determined by collecting ten replicate measurements on site-specific soil samples with metals concentrations 2 to 5 times the expected method detection limits. Based on these ten replicate measurements, a standard deviation on the replicate analysis was calculated. The method detection limits presented in Table 4 are defined as 3 times the standard deviation for each analyte.

The field-based method detection limits were generated by using the count times discussed earlier in this section. All the field-based method detection limits were calculated for soil samples that had been dried and ground and placed in a sample cup with the exception of the MAP Spectrum Analyzer. This instrument can only be operated in the *in situ* mode, meaning the samples were moist and not ground.

Some of the analytes such as cadmium, mercury, silver, selenium, and thorium were not detected or only detected at very low concentrations such that a field-based method detection limit could not be determined. These analytes are not presented in Table 4. Other analytes such as calcium, iron, potassium, and titanium were only found at high concentrations (thousands of mg/kg) so that reasonable method detection limits could not be calculated. These analytes also are not presented in Table 4.

13.5 Precision Measurements: The precision data is presented in Table 5. Each of the six FPXRF instruments performed 10 replicate measurements on 12 soil samples that had analyte concentrations ranging from nondetects to thousands of mg/kg. Each of the 12 soil samples underwent 4 different preparation techniques from *in situ* (no preparation) to dried and ground in a sample cup. Therefore, there were 48 precision data points for five of the instruments and 24 precision points for the MAP Spectrum Analyzer. The replicate measurements were taken using the source count times discussed at the beginning of this section.

For each detectable analyte in each precision sample a mean concentration, standard deviation, and RSD was calculated for each analyte. The data presented in Table 5 is an average RSD for the precision samples that had analyte concentrations at 5 to 10 times the MDL for that analyte for each instrument. Some analytes such as mercury, selenium, silver, and thorium were not detected in any of the precision samples so these analytes are not listed in Table 5. Some analytes such as cadmium, nickel, and tin were only detected at concentrations near the MDLs so that an RSD value calculated at 5 to 10 times the MDL was not possible.

One FPXRF instrument collected replicate measurements on an additional nine soil samples to provide a better assessment of the effect of sample preparation on precision. Table 6 shows these results. The additional nine soil samples were comprised of three from each texture and had analyte concentrations ranging from near the detection limit of the FPXRF analyzer to thousands of mg/kg. The FPXRF analyzer only collected replicate measurements from three of the preparation methods; no measurements were collected from the *in situ* homogenized samples. The FPXRF analyzer conducted five replicate measurements of the *in situ* field samples by taking measurements at five different points within the 4-inch by 4-inch sample square. Ten replicate measurements were collected for both the intrusive undried and unground and intrusive dried and ground samples contained in cups. The cups were shaken between each replicate measurement.

Table 6 shows that the precision dramatically improved from the *in situ* to the intrusive measurements. In general there was a slight improvement in precision when the sample was dried and ground. Two factors caused the precision for the *in situ* measurements to be poorer. The major factor is soil heterogeneity. By moving the probe within the 4-inch by 4-inch square, measurements of different soil samples were actually taking place within the square. Table 6 illustrates the dominant effect of soil heterogeneity. It overwhelmed instrument precision when the FPXRF analyzer was used in this mode. The second factor that caused the RSD values to be higher for the *in situ* measurements is the fact that only five versus ten replicates were taken. A lesser number of measurements caused the standard deviation to be larger which in turn elevated the RSD values.

13.6 Accuracy Measurements: Five of the FPXRF instruments (not including the MAP Spectrum Analyzer) analyzed 18 SRMs using the source count times and calibration methods given at the beginning of this section. The 18 SRMs included 9 soil SRMs, 4 stream or river sediment SRMs, 2 sludge SRMs, and 3 ash SRMs. Each of the SRMs contained known concentrations of certain target analytes. A percent recovery was calculated for each analyte in each SRM for each FPXRF instrument. Table 7 presents a summary of this data. With the exception of cadmium, chromium, and nickel, the values presented in Table 7 were generated from the 13 soil and sediment SRMs only. The 2 sludge and 3 ash SRMs were included for cadmium, chromium, and nickel because of the low or nondetectable concentrations of these three analytes in the soil and sediment SRMs.

Only 12 analytes are presented in Table 7. These are the analytes that are of environmental concern and provided a significant number of detections in the SRMs for an accuracy assessment. No data is presented for the X-MET 920 with the gas-filled proportional detector. This FPXRF instrument was calibrated empirically using site-specific soil samples. The percent recovery values from this instrument were very sporadic and the data did not lend itself to presentation in Table 7.

Table 8 provides a more detailed summary of accuracy data for one FPXRF instrument (TN 9000) for the 9 soil SRMs and 4 sediment SRMs. Table 8 shows the certified value, measured value, and percent recovery for five analytes. These analytes were chosen because they are of environmental concern and were most prevalently certified for in the SRM and detected by the FPXRF instrument. The first nine SRMs are soil and the last 4 SRMs are sediment. Percent recoveries for the four NIST SRMs were often between 90 and 110 percent for all analytes.

13.7 Comparability: Comparability refers to the confidence with which one data set can be compared to another. In this case, FPXRF data generated from a large study of six FPXRF instruments was compared to SW-846 Methods 3050 and 6010 which are the standard soil extraction for metals and analysis by inductively coupled plasma. An evaluation of comparability was conducted by using linear regression analysis. Three factors were determined using the linear regression. These factors were the y-intercept, the slope of the line, and the coefficient of determination (r^2).

As part of the comparability assessment, the effects of soil type and preparation methods were studied. Three soil types (textures) and four preparation methods were examined during the study. The preparation methods evaluated the cumulative effect of particle size, moisture, and homogenization on comparability. Due to the large volume of data produced during this study, linear regression data for six analytes from only one FPXRF instrument is presented in Table 9. Similar trends in the data were seen for all instruments.

Table 9 shows the regression parameters for the whole data set, broken out by soil type, and by preparation method. The soil types are as follows: soil 1--sand; soil 2--loam; and soil 3--silty clay. The preparation methods are as follows: preparation 1--in situ in the field; preparation 2--in situ, sample collected and homogenized; preparation 3--intrusive, with sample in a sample cup but sample still wet and not ground; and preparation 4--sample dried, ground, passed through a 40-mesh sieve, and placed in sample cup.

For arsenic, copper, lead, and zinc, the comparability to the confirmatory laboratory was excellent with r^2 values ranging from 0.80 to 0.99 for all six FPXRF instruments. The slopes of the regression lines for arsenic, copper, lead, and zinc, were generally between 0.90 and 1.00 indicating the data would need to be corrected very little or not at all to match the confirmatory laboratory data. The r^2 values and slopes of the regression lines for barium and chromium were

not as good as for the other for analytes, indicating the data would have to be corrected to match the confirmatory laboratory.

Table 9 demonstrates that there was little effect of soil type on the regression parameters for any of the six analytes. The only exceptions were for barium in soil 1 and copper in soil 3. In both of these cases, however, it is actually a concentration effect and not a soil effect causing the poorer comparability. All barium and copper concentrations in soil 1 and 3, respectively, were less than 350 mg/kg.

Table 9 shows there was a preparation effect on the regression parameters for all six analytes. With the exception of chromium, the regression parameters were primarily improved going from preparation 1 to preparation 2. In this step, the sample was removed from the soil surface, all large debris was removed, and the sample was thoroughly homogenized. The additional two preparation methods did little to improve the regression parameters. This data indicates that homogenization is the most critical factor when comparing the results. It is essential that the sample sent to the confirmatory laboratory match the FPXRF sample as closely as possible.

Section 11.0 of this method discusses the time necessary for each of the sample preparation techniques. Based on the data quality objectives for the project, an analyst must decide if it is worth the extra time required to dry and grind the sample for small improvements in comparability. Homogenization requires 3 to 5 minutes. Drying the sample requires one to two hours. Grinding and sieving requires another 10 to 15 minutes per sample. Lastly, when grinding and sieving is conducted, time must be allotted to decontaminate the mortars, pestles, and sieves. Drying and grinding the samples and decontamination procedures will often dictate that an extra person be on site so that the analyst can keep up with the sample collection crew. The cost of requiring an extra person on site to prepare samples must be balanced with the gain in data quality and sample throughput.

13.8 The following documents may provide additional guidance and insight on this method and technique:

13.8.1 Hewitt, A.D. 1994. "Screening for Metals by X-ray Fluorescence Spectrometry/Response Factor/Compton K_{α} Peak Normalization Analysis." *American Environmental Laboratory*. Pages 24-32.

13.8.2 Piorek, S., and J.R. Pasmore. 1993. "Standardless, In Situ Analysis of Metallic Contaminants in the Natural Environment With a PC-Based, High Resolution Portable X-Ray Analyzer." *Third International Symposium on Field Screening Methods for Hazardous Waste and Toxic Chemicals*. Las Vegas, Nevada. February 24-26, 1993. Volume 2, Pages 1135-1151.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical management for Waste Reduction* available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street N.W., Washington D.C. 20036, (202) 872-4477.

15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the American Chemical Society at the address listed in Sec. 14.2.

16.0 REFERENCES

1. Metorex. X-MET 920 User's Manual.
2. Spectrace Instruments. 1994. Energy Dispersive X-ray Fluorescence Spectrometry: An Introduction.
3. TN Spectrace. Spectrace 9000 Field Portable/Benchtop XRF Training and Applications Manual.
4. Unpublished SITE data, recieved from PRC Environment Management, Inc.

17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

The pages to follow contain Tables 1 through 9 and a method procedure flow diagram.

TABLE 1
INTERFERENCE FREE DETECTION LIMITS

Analyte	Chemical Abstract Series Number	Detection Limit in Quartz Sand (milligrams per kilogram)
Antimony (Sb)	7440-36-0	40
Arsenic (As)	7440-38-0	40
Barium (Ba)	7440-39-3	20
Cadmium (Cd)	7440-43-9	100
Calcium (Ca)	7440-70-2	70
Chromium (Cr)	7440-47-3	150
Cobalt (Co)	7440-48-4	60
Copper (Cu)	7440-50-8	50
Iron (Fe)	7439-89-6	60
Lead (Pb)	7439-92-1	20
Manganese (Mn)	7439-96-5	70
Mercury (Hg)	7439-97-6	30
Molybdenum (Mo)	7439-93-7	10
Nickel (Ni)	7440-02-0	50
Potassium (K)	7440-09-7	200
Rubidium (Rb)	7440-17-7	10
Selenium (Se)	7782-49-2	40
Silver (Ag)	7440-22-4	70
Strontium (Sr)	7440-24-6	10
Thallium (Tl)	7440-28-0	20
Thorium (Th)	7440-29-1	10
Tin (Sn)	7440-31-5	60
Titanium (Ti)	7440-32-6	50
Vanadium (V)	7440-62-2	50
Zinc (Zn)	7440-66-6	50
Zirconium (Zr)	7440-67-7	10

Source: References 1, 2, and 3

TABLE 2
SUMMARY OF RADIOISOTOPE SOURCE CHARACTERISTICS

Source	Activity (mCi)	Half-Life (Years)	Excitation Energy (keV)	Elemental Analysis Range	
Fe-55	20-50	2.7	5.9	Sulfur to Chromium Molybdenum to Barium	K Lines L Lines
Cd-109	5-30	1.3	22.1 and 87.9	Calcium to Rhodium Tantalum to Lead Barium to Uranium	K Lines K Lines L Lines
Am-241	5-30	458	26.4 and 59.6	Copper to Thulium Tungsten to Uranium	K Lines L Lines
Cm-244	60-100	17.8	14.2	Titanium to Selenium Lanthanum to Lead	K Lines L Lines

Source: Reference 1, 2, and 3

TABLE 3
SUMMARY OF X-RAY TUBE SOURCE CHARACTERISTICS

Anode Material	Recommended Voltage Range (kV)	K-alpha Emission (keV)	Elemental Analysis Range	
Cu	18-22	8.04	Potassium to Cobalt Silver to Gadolinium	K Lines L Lines
Mo	40-50	17.4	Cobalt to Yttrium Europium to Radon	K Lines L Lines
Ag	50-65	22.1	Zinc to Technicium Ytterbium to Neptunium	K Lines L Lines

Source: Reference 4

Notes: The sample elements excited are chosen by taking as the lower limit the same ratio of excitation line energy to element absorption edge as in Table 2 (approximately 0.45) and the requirement that the excitation line energy be above the element absorption edge as the upper limit (L2 edges used for L lines). K-beta excitation lines were ignored.

TABLE 4
FIELD-BASED METHOD DETECTION LIMITS (mg/kg)^a

Analyte	Instrument					
	TN 9000	TN Lead Analyzer	X-MET 920 (SiLi Detector)	X-MET 920 (Gas-Filled Detector)	XL Spectrum Analyzer	MAP Spectrum Analyzer
Antimony	55	NR	NR	NR	NR	NR
Arsenic	60	50	55	50	110	225
Barium	60	NR	30	400	NR	NR
Chromium	200	460	210	110	900	NR
Cobalt	330	NR	NR	NR	NR	NR
Copper	85	115	75	100	125	525
Lead	45	40	45	100	75	165
Manganese	240	340	NR	NR	NR	NR
Molybdenum	25	NR	NR	NR	30	NR
Nickel	100	NR	NA	NA	NA	NR
Rubidium	30	NR	NR	NR	45	NR
Strontium	35	NR	NR	NR	40	NR
Tin	85	NR	NR	NR	NR	NR
Zinc	80	95	70	NA	110	NA
Zirconium	40	NR	NR	NR	25	NR

Source: Reference 4

^a MDLs are related to the total number of counts taken. See Section 13.3 for count times used to generate this table.

NR Not reported.

NA Not applicable; analyte was reported but was not at high enough concentrations for method detection limit to be determined.

**TABLE 5
PRECISION**

Analyte	Average Relative Standard Deviation for Each Instrument at 5 to 10 Times the MDL					
	TN 9000	TN Lead Analyzer	X-MET 920 (SiLi Detector)	X-MET 920 (Gas-Filled Detector)	XL Spectrum Analyzer	MAP Spectrum Analyzer
Antimony	6.54	NR	NR	NR	NR	NR
Arsenic	5.33	4.11	3.23	1.91	12.47	6.68
Barium	4.02	NR	3.31	5.91	NR	NR
Cadmium	29.84 ^a	NR	24.80 ^a	NR	NR	NR
Calcium	2.16	NR	NR	NR	NR	NR
Chromium	22.25	25.78	22.72	3.91	30.25	NR
Cobalt	33.90	NR	NR	NR	NR	NR
Copper	7.03	9.11	8.49	9.12	12.77	14.86
Iron	1.78	1.67	1.55	NR	2.30	NR
Lead	6.45	5.93	5.05	7.56	6.97	12.16
Manganese	27.04	24.75	NR	NR	NR	NR
Molybdenum	6.95	NR	NR	NR	12.60	NR
Nickel	30.85 ^a	NR	24.92 ^a	20.92 ^a	NA	NR
Potassium	3.90	NR	NR	NR	NR	NR
Rubidium	13.06	NR	NR	NR	32.69 ^a	NR
Strontium	4.28	NR	NR	NR	8.86	NR
Tin	24.32 ^a	NR	NR	NR	NR	NR
Titanium	4.87	NR	NR	NR	NR	NR
Zinc	7.27	7.48	4.26	2.28	10.95	0.83
Zirconium	3.58	NR	NR	NR	6.49	NR

Source: Reference 4

^a These values are biased high because the concentration of these analytes in the soil samples was near the detection limit for that particular FPXRF instrument.

NR Not reported.

NA Not applicable; analyte was reported but was below the method detection limit.

TABLE 6
PRECISION AS AFFECTED BY SAMPLE PREPARATION

Analyte	Average Relative Standard Deviation for Each Preparation Method		
	In Situ-Field	Intrusive- Undried and Unground	Intrusive- Dried and Ground
Antimony	30.1	15.0	14.4
Arsenic	22.5	5.36	3.76
Barium	17.3	3.38	2.90
Cadmium ^a	41.2	30.8	28.3
Calcium	17.5	1.68	1.24
Chromium	17.6	28.5	21.9
Cobalt	28.4	31.1	28.4
Copper	26.4	10.2	7.90
Iron	10.3	1.67	1.57
Lead	25.1	8.55	6.03
Manganese	40.5	12.3	13.0
Mercury	ND	ND	ND
Molybdenum	21.6	20.1	19.2
Nickel ^a	29.8	20.4	18.2
Potassium	18.6	3.04	2.57
Rubidium	29.8	16.2	18.9
Selenium	ND	20.2	19.5
Silver ^a	31.9	31.0	29.2
Strontium	15.2	3.38	3.98
Thallium	39.0	16.0	19.5
Thorium	NR	NR	NR
Tin	ND	14.1	15.3
Titanium	13.3	4.15	3.74
Vanadium	NR	NR	NR
Zinc	26.6	13.3	11.1
Zirconium	20.2	5.63	5.18

Source: Reference 4

^a These values may be biased high because the concentration of these analytes in the soil samples was near the detection limit.

ND Not detected.

NR Not reported.

**TABLE 7
ACCURACY**

Analyte	Instrument															
	TN 9000				TN Lead Analyzer				X-MET 920 (SiLi Detector)				XL Spectrum Analyzer			
	n	Range of % Rec.	Mean % Rec.	SD	n	Range of % Rec.	Mean % Rec.	SD	n	Range of % Rec.	Mean % Rec.	SD	n	Range of % Rec.	Mean % Rec.	SD
Sb	2	100-149	124.3	NA	--	--	--	--	--	--	--	--	--	--	--	--
As	5	68-115	92.8	17.3	5	44-105	83.4	23.2	4	9.7-91	47.7	39.7	5	38-535	189.8	206
Ba	9	98-198	135.3	36.9	--	--	--	--	9	18-848	168.2	262	--	--	--	--
Cd	2	99-129	114.3	NA	--	--	--	--	6	81-202	110.5	45.7	--	--	--	--
Cr	2	99-178	138.4	NA	--	--	--	--	7	22-273	143.1	93.8	3	98-625	279.2	300
Cu	8	61-140	95.0	28.8	6	38-107	79.1	27.0	11	10-210	111.8	72.1	8	95-480	203.0	147
Fe	6	78-155	103.7	26.1	6	89-159	102.3	28.6	6	48-94	80.4	16.2	6	26-187	108.6	52.9
Pb	11	66-138	98.9	19.2	11	68-131	97.4	18.4	12	23-94	72.7	20.9	13	80-234	107.3	39.9
Mn	4	81-104	93.1	9.70	3	92-152	113.1	33.8	--	--	--	--	--	--	--	--
Ni	3	99-122	109.8	12.0	--	--	--	--	--	--	--	--	3	57-123	87.5	33.5
Sr	8	110-178	132.6	23.8	--	--	--	--	--	--	--	--	7	86-209	125.1	39.5
Zn	11	41-130	94.3	24.0	10	81-133	100.0	19.7	12	46-181	106.6	34.7	11	31-199	94.6	42.5

Source: Reference 4

n Number of samples that contained a certified value for the analyte and produced a detectable concentration from the FPXRF instrument.
SD Standard deviation.
NA Not applicable; only two data points, therefore, a SD was not calculated.
%Rec. Percent recovery.
-- No data.

TABLE 8
ACCURACY FOR TN 9000^a

Standard Reference Material	Arsenic			Barium			Copper			Lead			Zinc		
	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.
RTC CRM-021	24.8	ND	NA	586	1135	193.5	4792	2908	60.7	144742	149947	103.6	546	224	40.9
RTC CRM-020	397	429	92.5	22.3	ND	NA	753	583	77.4	5195	3444	66.3	3022	3916	129.6
BCR CRM 143R	--	--	--	--	--	--	131	105	80.5	180	206	114.8	1055	1043	99.0
BCR CRM 141	--	--	--	--	--	--	32.6	ND	NA	29.4	ND	NA	81.3	ND	NA
USGS GXR-2	25.0	ND	NA	2240	2946	131.5	76.0	106	140.2	690	742	107.6	530	596	112.4
USGS GXR-6	330	294	88.9	1300	2581	198.5	66.0	ND	NA	101	80.9	80.1	118	ND	NA
NIST 2711	105	104	99.3	726	801	110.3	114	ND	NA	1162	1172	100.9	350	333	94.9
NIST 2710	626	722	115.4	707	782	110.6	2950	2834	96.1	5532	5420	98.0	6952	6476	93.2
NIST 2709	17.7	ND	NA	968	950	98.1	34.6	ND	NA	18.9	ND	NA	106	98.5	93.0
NIST 2704	23.4	ND	NA	414	443	107.0	98.6	105	106.2	161	167	103.5	438	427	97.4
CNRC PACS-1	211	143	67.7	--	772	NA	452	302	66.9	404	332	82.3	824	611	74.2
SARM-51	--	--	--	335	466	139.1	268	373	139.2	5200	7199	138.4	2200	2676	121.6
SARM-52	--	--	--	410	527	128.5	219	193	88.1	1200	1107	92.2	264	215	81.4

Source: Reference 4

^a All concentrations in milligrams per kilogram.
 %Rec. Percent recovery.
 ND Not detected.
 NA Not applicable.
 -- No data.

TABLE 9
REGRESSION PARAMETERS FOR COMPARABILITY¹

	Arsenic				Barium				Copper			
	n	r ²	Int.	Slope	n	r ²	Int.	Slope	n	r ²	Int.	Slope
All Data	824	0.94	1.62	0.94	1255	0.71	60.3	0.54	984	0.93	2.19	0.93
Soil 1	368	0.96	1.41	0.95	393	0.05	42.6	0.11	385	0.94	1.26	0.99
Soil 2	453	0.94	1.51	0.96	462	0.56	30.2	0.66	463	0.92	2.09	0.95
Soil 3	—	—	—	—	400	0.85	44.7	0.59	136	0.46	16.60	0.57
Prep 1	207	0.87	2.69	0.85	312	0.64	53.7	0.55	256	0.87	3.89	0.87
Prep 2	208	0.97	1.38	0.95	315	0.67	64.6	0.52	246	0.96	2.04	0.93
Prep 3	204	0.96	1.20	0.99	315	0.78	64.6	0.53	236	0.97	1.45	0.99
Prep 4	205	0.96	1.45	0.98	313	0.81	58.9	0.55	246	0.96	1.99	0.96

	Lead				Zinc				Chromium			
	n	r ²	Int.	Slope	n	r ²	Int.	Slope	n	r ²	Int.	Slope
All Data	1205	0.92	1.66	0.95	1103	0.89	1.86	0.95	280	0.70	64.6	0.42
Soil 1	357	0.94	1.41	0.96	329	0.93	1.78	0.93	—	—	—	—
Soil 2	451	0.93	1.62	0.97	423	0.85	2.57	0.90	—	—	—	—
Soil 3	397	0.90	2.40	0.90	351	0.90	1.70	0.98	186	0.66	38.9	0.50
Prep 1	305	0.80	2.88	0.86	286	0.79	3.16	0.87	105	0.80	66.1	0.43
Prep 2	298	0.97	1.41	0.96	272	0.95	1.86	0.93	77	0.51	81.3	0.36
Prep 3	302	0.98	1.26	0.99	274	0.93	1.32	1.00	49	0.73	53.7	0.45
Prep 4	300	0.96	1.38	1.00	271	0.94	1.41	1.01	49	0.75	31.6	0.56

Source: Reference 4

- ¹ Log-transformed data
n Number of data points
r² Coefficient of determination
Int. Y-intercept
— No applicable data

METHOD 6200

FIELD PORTABLE X-RAY FLUORESCENCE SPECTROMETRY FOR THE DETERMINATION OF ELEMENTAL CONCENTRATIONS IN SOIL AND SEDIMENT

